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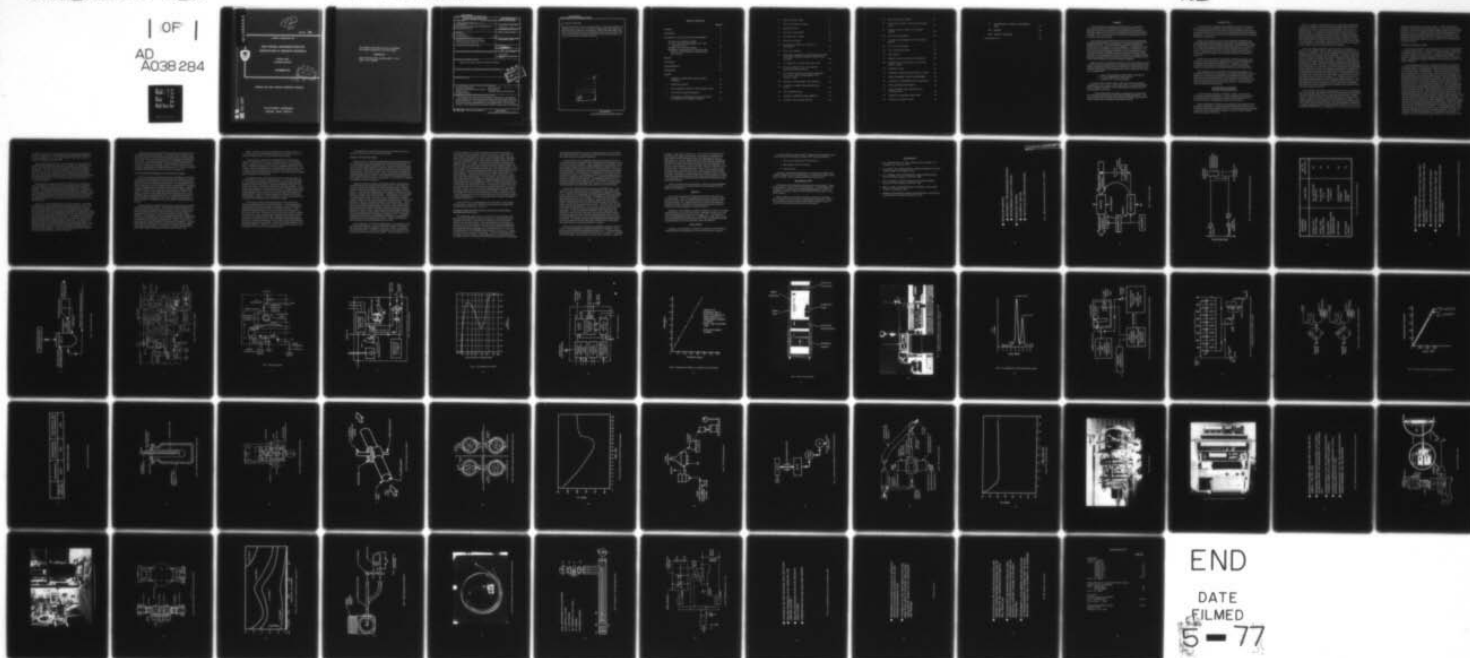
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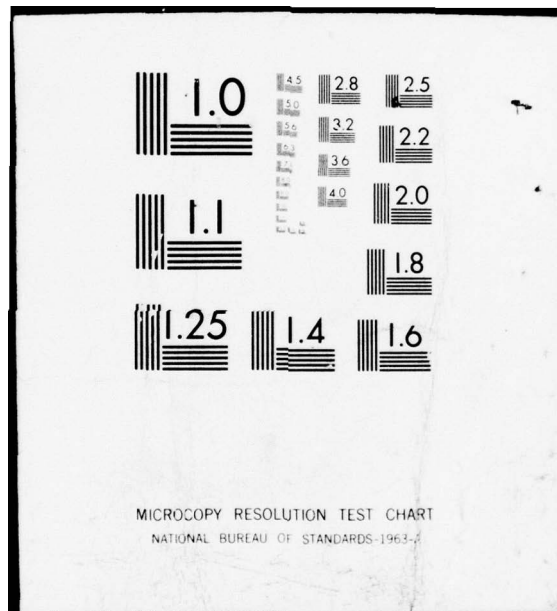
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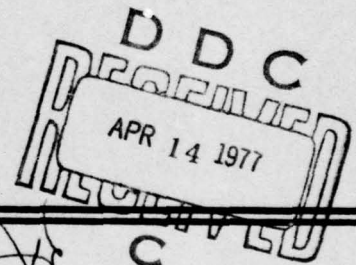
TECHNICAL MEMORANDUM 2222

NEW CONTROL INSTRUMENTATION FOR  
MANUFACTURE OF ENERGETIC MATERIALS



MICHAEL HALIK  
CLEOPHUS MCINTOSH

DECEMBER 1976



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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER Technical Memorandum 2222 ✓	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) New Control Instrumentation for Manufacture of Energetic Materials		5. TYPE OF REPORT & PERIOD COVERED Interim Report
7. AUTHOR(s) Michael Halik Cleophus McIntosh		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Manufacturing Technology Directorate Picatinny Arsenal, Dover, NJ ✓		8. CONTRACT OR GRANT NUMBER(s) PA-TM-2222
11. CONTROLLING OFFICE NAME AND ADDRESS		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE December 1976
		13. NUMBER OF PAGES 65
		15. SECURITY CLASS. (of this report) Unclassified
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Liquid chromatography      Open-closed loop Ultraviolet spectrophotometric analyzer      Acid analyzer Feedback loops      Automatic sampling instrumentation Energetic materials		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Prototype process controls with fast detection, measurement, and feedback control capability have been developed by Picatinny Arsenal, Radford AAP, and selected contractors. These prototype on-stream process analyzers will perform the function now being performed by various laboratory tests. The basic difference is in the method used for sampling, detection, measurement, and feedback.		

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20. Abstract (continued)

Sampling will be done on-stream wherever possible. Under these conditions, no sample retrieval is necessary. By utilizing the latest technological advances in physicochemical sensors and instrumentation, detection and measurement of the process control parameters can be accomplished in real time in most cases. Outputs from the detection and measuring instruments can be used to provide corrective action by means of automatic feedback.

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## SUMMARY

In the modernization of ammunition production it is desirable to incorporate automatic instrumentation which can perform on-line measurements of critical-process stream parameters and generate proportional signals that can be used to automatically control the measured parameter. This is especially true in the case of automated and continuous manufacturing processes.

This technical memorandum describes new control instrumentation designed and developed for application to the automated manufacture of energetic materials. This instrumentation provides fast detection, measurement, and feedback for control capability. Sampling is done on-stream wherever possible so that sample retrieval is unnecessary. By utilizing the latest technological advances in physicochemical sensors and instrumentation, detection and measurement of the process control parameters is accomplished in real time in most cases. Outputs from the detection and measuring instruments are utilized for performing any corrective action required by means of automatic feedback.

Specific control instrumentation covered in this memorandum includes systems for both the continuous TNT manufacturing process and the continuous automated explosives melt-pour operation. A description is presented for the design and development of the following on-line TNT analyzers at Radford AAP.

1. Liquid chromatographic  $\text{H}_2\text{SO}_4/\text{HNO}_3$  ratio analyzer
2. UV spectrophotometric  $\text{HNO}_3$  analyzer
3. Automatic TNT set-point analyzer.

In order to meet a specific safety requirement for the continuous automated melt-pour system, a fiber-optic sensor was developed which detects leakage of explosive past the diaphragm of a pump utilized for pumping molten explosives.

The instrumentation developed is applicable to all continuous TNT manufacturing lines and explosives melt-loading processes. The same concepts and techniques can be applied to similar control requirements in other areas of energetic materials processing.

## INTRODUCTION

The modernization of ammunition production requires the incorporation of automatic instrumentation that possesses the ability to perform on-line measurements of critical process stream parameters and generate proportional signals that can be used to automatically control the measured parameter. This is especially true in the case of automated and continuous manufacturing processes.

Under the first phase of a multi-year Manufacturing Methods and Technology program to automate the continuous TNT production facility process controls, sufficient data was generated to produce design criteria for a liquid chromatographic  $\text{H}_2\text{SO}_4/\text{HNO}_3$  ratio analyzer, an ultraviolet spectrophotometric  $\text{HNO}_3$  analyzer, and an automatic TNT set-point system. The design criteria were used for the procurement of each of these systems with automatic on-line sample retrieval and preparation capability.

Under the second phase of the program, prototype analyzers and controls have been procured, installed, and are currently undergoing debugging and evaluation at Radford AAP. In addition, a molten explosives pump malfunction detection and control system has been designed and procured for the automated explosive melt load process.

Upon completion of the evaluation, the systems developed for the continuous TNT process will be ready for permanent installation at either Radford or Volunteer AAP, depending on the operational status of the TNT process lines at these facilities. The explosives pump malfunction detection and control system is currently installed in the automated high explosive melt-pour pilot line at Picatinny Arsenal. Further systems will be installed at Lone Star AAP.

## DEVELOPMENT OF PROCESS CONTROL INSTRUMENTATION

The two general categories of automatic control, open loop and closed loop, are shown in Figure 1. Each is characterized by whether or not measurement information is fed back from the process to an automatic controller programmed to take corrective action.

Open-loop automatic control does not feed back information from the process to a controller. A familiar example of this type of control is the automatic home washing machine which is electromechanically programmed to control a series of operations necessary to wash a load of clothing based solely upon a time cycle.

Figure 2 illustrates closed-loop control in simple block diagram form. In this case, information in the form of a measurement signal is obtained regarding the process, or process control parameter, by the use of a suitable sensor. This signal is fed by the transmitter as an input to the analog controller. The controller contains an error-detecting circuit which compares this input signal with a correlated, internally generated signal representing the desired parameter value or set point; any difference between these two signals causes the controller to generate a corrective output signal. This signal is applied to the final control element which, in turn, adjusts a variable input to the process in such a way as to return the controlled variable to the desired process condition.

The use of a digital computer in process control is illustrated in Figure 3. The computer takes the place of the analog controller shown in the previous illustration. The control computer activates the final control element to make the necessary process adjustment. A process control computer has the ability to handle many control loops and therefore takes the place of a number of individual controllers. In addition to replacing the controller in the closed-loop control scheme, the process control computer has the ability to make complex computations regarding the interactions of the process variables. Therefore, if sufficient data is available regarding these interactions, and if this data can be used to develop a reliable mathematical model of the process, the computer can optimize the production process provided that accurate measurements of the process parameters can be inputted into the computer on a timely basis. In this presentation we are concerned only with that section of the closed-loop control which involves the measurement of the process variable, i.e., that part of the control loop consisting of the sensor with the associated signal conditioner. More specifically, MTD's efforts have been devoted in this area exclusively to those particular sensors which have a critical application to automatic remote control of explosives manufacture but which are not available off-the-shelf from commercial vendors.

As an example, with regard to the production of TNT, its manufacture has been up-graded and modernized from the original batch process dating back to the period of WW II to the current continuous operation. However, in order to maintain integrity of the continuous process, the parameters shown in Figure 4 are still measured by old laboratory techniques. In fact, a small laboratory area is located in each TNT production building where some of these measurements are made. After each parameter is measured, an operator manipulates the final control element manually (in the case of the Radford AAP operation) or automatically (in the case of the more recent



computer-controlled line at Volunteer AAP). In 1974 a program was initiated by MTD at Picatinny Arsenal to design, construct, and evaluate automatic instrumental systems capable of measuring these TNT process parameters on line. In addition to the TNT control instrumentation, an explosives-pump malfunction detection system was developed for the automated high explosive melt-pour line. Figure 5 presents the instrumentation currently developed for process control in the manufacture of energetic materials.

#### UV Nitric Acid Analyzer System

Nitric acid concentration is one of the most critical control parameters in the manufacture of TNT. A typical TNT nitration stage is illustrated in schematic diagram form in Figure 6. A UV nitric acid analyzer to control  $\text{HNO}_3$  nitrator concentration was developed for this purpose. A schematic diagram of the total automatic on-line UV Nitric Acid Analyzer System, including the sampling, photometric and electronic sections, is shown in Figure 7.

The sampling system (Fig 8) is designed to deliver a cleaned and diluted acid sample from the process stream to the analyzer section on a continuous basis. The acid sample is metered into the sample stream by a metering pump. Simultaneously, dilution water enters the sample system through a filter and de-ionizer, through a heater to bring the water temperature up to the acid temperature (to keep nitro bodies from being precipitated) and through a flow regulator to a mixing elbow. By adjusting the acid pump flow and the water flow, the required acid dilution can be obtained. The diluted sample acid is then fed to a cooler-separator where it is cooled to approximately  $35^\circ\text{C}$ . At this temperature the aqueous and oil/solid phases are separated. A small quantity of the diluted, separated sample (approximately 8cc/min) is pumped through an activated charcoal filter. The charcoal filters are used to absorb residual nitro bodies that may remain dissolved in the sample stream. Nitro bodies absorb heavily in the UV spectral region, and they must therefore be eliminated from the sample stream; otherwise their presence would interfere with the measurement. Eight solenoid-switched, activated-charcoal filters are used in a parallel configuration. An electronic switching circuit operates the solenoid valves to direct the acid flow to consecutive filters at predetermined sequential time intervals. After each filter has been used, the sequence is repeated. Light indicators show which filter is being used at any given time; also, a particular filter or a new sequence starting point can also be selected manually. From the charcoal filter, the acid sample passes to a two-position rotary stream-selector valve which directs either the sample acid or calibration acid to



the analyzer alternately for approximately 10-minute and 5-minute time periods, respectively. This technique permits instantaneous detection of any instrument failure. The rotary stream-selector valve is pneumatically actuated by solenoid valves.

The UV spectrometer section is shown in Figure 9. The ultraviolet radiation from the deuterium lamp passes through a monochromator consisting of the two ultraviolet interference filters positioned 180° apart on the rotor. The rotor revolves at 3900 rpm, which gives a signal frequency of 65 cycles per second. One of the filters has its peak transmission at 300 nm, corresponding to the maximum absorption of  $\text{HNO}_3$ , while the second filter transmits at 360 nm, at which wavelength the  $\text{HNO}_3$  has a minimal absorbance. The ultraviolet spectrum of nitric acid is presented in Figure 10.

After passing through the optical filters (Fig 9), the radiation traverses the sample cell, which has a 3 mm pathlength, and is ultimately focused on the photomultiplier detector tube containing an integral pre-amplifier. As the sample passes through the sample cell, its absorption is measured alternately at both the 300 nm and 360 nm wavelengths. This dual wavelength absorption technique enables the analysis to be independent of the absolute absorption, and hence, insensitive to solid or other broadband contaminants, as well as to variations in the UV source or detector elements.

Figure 11 is a block diagram of the electronics module. The module LA-O/A contains the signal amplifier and automatic gain control circuits. The AGC circuit operates on the reference-signal pulse only, to maintain the reference pulse at a constant level and adjust the total amplification accordingly. This technique compensates for any variations occurring in the optics or sample that would tend to affect the true  $\text{HNO}_3$  measurement. The conditioned signals go to module LAA11. This module serves to subtract the sample signal pulse from the reference signal pulse to produce a signal proportional to the  $\text{HNO}_3$  sample concentration. In addition, this module separates the calibration signal from the sample acid signal. A recorder output is provided from the calibration acid signal while a 10-50 MA sample signal output is provided for a standard analog process controller or digital computer. A curve relating the absorbance of  $\text{HNO}_3$  as a function of concentration is shown in Figure 12.

Tests have been run on the nitric acid analyzer to indicate its long term stability and reproducibility. Variations in signal output over a few hours time were within 5% of the full scale range, or within 0.005% of the actual nitric acid concentration of the sample passing through the photometer. To bring the measurement for a 15%  $\text{HNO}_3$  acid within the 0.1% to 0.2% range of the analyzer, the dilution factor would be 100:1. Therefore, the accuracy would be within 0.5% of the actual acid concentration. A photograph of the  $\text{HNO}_3$  analyzer which is currently installed at Radford AAP for extensive testing is shown in Figure 13.

#### On-Line Liquid Chromatographic (LC) Acid Analysis System

Figure 14 illustrates the laboratory bench (breadboard) installation of the Liquid Chromatographic (LC) System that was originally used in performing off-line evaluation to determine the LC system's capability to perform the required TNT/acid ratio analysis. This breadboard installation is shown in order to illustrate the basic components of an analytical liquid chromatograph which include a high-pressure pump, a packed separation column which is the heart of the system, a sample injection septum, a detector, and a signal-processing electronic section. The pump is a positive displacement metering type identical to that used in the  $\text{HNO}_3$  analyzer discussed previously. The pump passes the carrier liquid through the separation column under high pressure (250 psi). Either Porapak-S or Sephadex G-10 can be used for separation packing material.

The acid sample is injected into the carrier liquid stream just before it enters the separation column. The separation column separates the nitric acid from the sample mixture by virtue of the fact that the column packing material retains the nitric acid selectively on its surface for a finite period of time while the sulfuric acid passes through without hindrance. The detector, which is essentially an electrical conductivity bridge, detects the passage of each acid and measures the concentration as a function of the respective conductivities. Figure 14 also shows a signal amplifier, signal conditioner, and electronic integrator. Actually, the concentration of the acids is a function of the area under the peak, rather than the peak height. The electronic integrator performs the required peak integration automatically and prints out each value.

Figure 15 shows a typical chromatogram for a sample mixture. It can be seen from this chromatogram that the sulfuric acid is the first to transit the column since it was not retained on the column.

The prototype LC System in block diagram form is presented in Figure 16, while Figure 17 shows a schematic of the on-line sampling system. Sample acids are fed from each TNT nitrator section by gravity feed. Automatically operated valves permit sequential sampling of each nitrator. Most of the sample acid is recycled back into the process, while a small portion is metered out into a permeation cell where it is mixed with water and the nitro bodies are removed. The treated sample is passed onto the separation column section.

The operation of the acid-sample loop valve is shown in Figure 18. In load position, the sample waiting analysis is in a flow-through status so as to flush the sampling system while the previous sample is passing through the separation column. When the chromatogram has been completed, the valve is automatically advanced to the inject position. In this position the sample loop is switched from the flow-through position to the sample-inject position. This position now aligns the sample loop with the chromatographic column and the carrier fluid, effectively injecting the sample acid into the carrier liquid to begin the preparation of the next chromatogram. Meanwhile, the next sample is in a flow-through position flushing out the sampling system with the next sample.

The electronic signal-processing section (Fig 14) performs the necessary integration and acid ratio calculations. The chromatogram is used to control the electronic signal processing. As the chromatogram is developed its derivative function is constantly monitored. As programmed, integration begins at the point the derivative changes from zero to a preset value, continues until such time as the derivative passes through the zero slope at the peak maximum and again reaches a second zero value at the end of the first peak. Integration of the second peak proceeds in an identical fashion. When both peaks have been integrated, their ratio is obtained electronically and a control signal proportioned to the  $\text{HNO}_3/\text{H}_2\text{SO}_4$  ratio is generated. Printout of the chromatogram is also provided on demand. Figure 19 is a graph relating  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$  concentrations as a function of the integration count.

The coefficient of variation (Fig 20) for the instrumental system is about half the current off-line titration technique.

#### Automatic TNT Set-Point System

Figure 21 shows a Standard Solidification Point System and Figure 22 a cross-sectional view of the Automatic TNT Set-Point System which is currently being evaluated at Radford AAP. Its essential components consist of the TNT sample chamber, a vibratory means to vibrate the sample during cooling, and a quartz thermometer that measures the sample temperature with a resolution of  $0.01^{\circ}\text{C}$ . Ports are provided for cooling the sample chamber with air during the cooling cycle and for reheating with steam after completion of the test. The mechanical sampling principle for the instrument is basically that of a plug valve. A view of the roto-sampler test chamber is shown in Figure 23.

Figure 24 shows the position of the sample chamber during the four indexed phases of the set-point determination cycle. In the initial position (A, Fig 24) the chamber is in a flow-through position. Steam is passed around the sample chamber to maintain its temperature at  $100^{\circ}\text{C}$ . In the second indexed position (B, Fig 24), a 180-g slug of TNT sample is isolated in the sample chamber. Simultaneously, air is passed around the sample chamber at a fast rate. In this position, the sample is in a rapid cooling status. Vibratory energy is applied to the cooling sample to induce crystallization and minimize super-cooling. In the third position (C, Fig 24), the sample chamber is in the  $90^{\circ}$  position. The air flow rate is reduced to decrease the cooling rate. The actual set point is determined when the sample chamber is in this position. After the set point has been determined, the sample chamber is indexed to the  $135^{\circ}\text{C}$  position for remelt. Steam is again passed around the sample chamber to remelt the crystallized portion of the TNT prior to indexing to the  $180^{\circ}$  flow-through position for the beginning of another cycle. The sampler is automatically controlled by a dedicated minicomputer. Its operation is controlled on a time and cooling-curve basis. Figure 28 illustrates a typical cooling curve relating temperature with time.

A flow diagram of the TNT finishing operation, into which the initial system will be installed, is shown in Figure 26. Figure 27 illustrates the installation of the roto-sampler in the main TNT line running from the drier to the flaker. A half-inch coaxial pass line is used to divert a portion of the flow through the roto-sampler. In actual operation, two



set-point samplers will be used in parallel as indicated in this figure. A drawing of a set-point sampler is shown in Figure 28. During start-up of the system, both samplers will be in a flow-through status. The computer will read the temperature of the samples in each sampler alternately. As initially programmed, a set-point cycle will not begin unless the temperature of the TNT is 100°C. If both samplers indicate a temperature of 100°C or higher, the computer will pick a sampler at random to commence the cycle, while the other sampler remains in a flow-through position. The active sampler will be indexed to the fast cool or 45° sample chamber position. The vibrator will be activated automatically when the sample chamber is in the 45° position. When the temperature of the sample reaches 86°C, the sample chamber is indexed to the 90° position; slow cooling occurs at this time. When the sample chamber is in this position, the computer begins to measure the slope of the cooling curve being generated while at the same time it keeps track of the temperature. When the computer measures a zero slope condition for a prescribed period of time, it prints out the temperature as the set point for that sample of TNT. If the measured temperature is out of the programmed limits for the desired set-point temperature, an alarm is activated. In any case, once a set-point temperature has been determined, the second sampler is activated while the first sampler is indexed to the 135° position for remelt for a prescribed period of time and then to the 180°, or flow-through position, to wait for the next cycle while the second sampler is operated through its cycle. A cooling curve for a vibrated TNT sample is shown in Figure 29.

Figures 30 and 31 are photographs of the roto-sampler and computer-control system, respectively. Benefits derived from the developed TNT instrumentation are shown in Figure 32.

#### **Pulsafeeder Pump Malfunction Detection and Control In High Explosive Automated Melt-Pour Process**

The Pulsafeeder pump which is in current use in the automated melt-pour pilot line at Picatinny is shown in Figure 33. This pump is used to transport molten TNT and Composition B explosives from a continuous melter to the volumetric loader. The essential elements of this pump include the positive displacement piston which works against the diaphragm through a hydraulic oil coupling. Pressure pulses are transmitted to the pump head through the diaphragm and a second hydraulic oil. Figure 34 is a photograph of the Pulsafeeder pump installed in the pilot line. Figure 35 shows a cross section of the pump head. It is this section that performs the actual explosive pumping. The pumping action is accomplished when the pressure pulses, transmitted from the piston through the first diaphragm



and through the hydraulic oil, exert a squeezing action on the hydratube. This squeezing action, in combination with the ball-check valves, forces the molten explosive through the hydratube.

During operation of the automated explosives melt-pour pilot line, it was learned that the hydratube was the weakest link in the pumping system in terms of long term reliability. After extended use the hydratube, which was originally constructed of a polyfluoroelastomeric rubber, would develop cracks causing seepage of explosive into the hydraulic oil surrounding the hydratube. Figure 36 shows a spectogram relating the absorbance of the hydraulic oil contaminated with Composition B as a function of light radiation wavelength. The absorbance spectra of oil containing varying amounts of Composition B are illustrated. It is noted that the peak absorption occurs at approximately 500 nm. Unadulterated hydraulic oil was found not to absorb in this spectral region. A change to a silicone rubber hydratube extended its operational life. However, it was considered prudent to develop a hydratube malfunction detection and control system.

Figure 37 illustrates the developed system in schematic form. It consists of a visible radiation source, a bifurcated fiber-optic light guide with a reflective sensing end and two photosensors arranged in a bridge circuit. Figure 38 is a photograph of the bifurcated fiber-optic light guide and the elbow into which it is mounted. A schematic of the malfunction detector optical path is shown in Figure 39. Light from the source is passed through optical filters that isolate a band of light in the 500 nm region. The optical filters are used to optimize the sensitivity of the detection system to the presence of TNT in the oil. From the filters the light is focused on the photodetectors; in case of  $D_1$ , directly, and in the case of  $D_2$ , after the light has traversed through the bifurcated fiber-optic light guide. In traversing the bifurcated fiber-optic light guide, the light enters one arm of the light guide, passes through the main section and into the hydraulic field. After passing through the fluid, the light is reflected back into the fiber-optic light guide and focused on  $D_2$  by the second arm. This scheme permits a remote measurement of light absorption of the hydraulic fluid by locating the sensing end of the fiber-optic light guide directly in the pump hydraulic oil circuit.

Figure 40 is a schematic drawing of the detection instrument's electric circuit. The photodetectors are actually photoresistive elements. They form two arms of a standard Wheatstone bridge circuit. The potentiometer  $R_2$  forms the other two arms of the detection circuit. Initially  $R_2$  is adjusted so that the bridge is balanced and no current flows through  $R_1$ ; under this

condition, meter relay M reads zero. This is the normal state when  $D_1$  is looking at the light source directly, and when  $D_2$  is looking at the light that has passed through the fiber-optic light guide and the unadulterated oil. If a leak should develop in the hydratube, leakage of the explosive into the oil would cause some of the light passing through the fiber-optic light guide (and the hydraulic oil) to be absorbed. Detector  $D_2$  would therefore receive less light, causing an imbalance in the bridge circuit. The bridge imbalance would, in turn, cause a current to flow through  $R_1$  and the meter relay. The current passing through the meter relay would in turn activate the 24-volt power relay, cutting the power to the pump and shutting down the explosive pump as a result. Tests of the detection system have shown that a concentration of 0.5% TNT in the hydraulic oil will cause sufficient absorption to activate the pump shut-down sequence.

Figure 41 outlines the instrumentation currently in the development stage; while Figures 42A and 42B summarize the development of new control instrumentation.

### BENEFITS

The current procedures for obtaining analytical process data are noninstrumental. They are outmoded by present day standards, costly to perform, and require a chemical laboratory facility within the TNT process building. It is estimated that the new control instrumentation program will make it possible to replace most of the chemical process-control tests with instrumental on-stream analyzers.

Installation of all automatic instrument controls will result in a savings of \$203,500 per year in labor costs alone. Additional savings will result from the reduced need to rework "out-of-spec" TNT. This estimate is based on the Radford AAP operation. Additionally, proportional savings will be effected as additional continuous TNT lines instrumented with the developed analyzers go into operation at other installations.

### CONCLUSIONS

Automatic instrumentation is necessary to perform on-line control of critical-process stream parameters and to maintain process integrity.

On-stream analyzers can be used to replace chemical laboratory type control testing of the following continuous TNT process parameters:

1. Nitric acid concentration in TNT nitrators
2.  $\text{HNO}_3/\text{H}_2\text{SO}_4$  ratio TNT nitrators
3. TNT set points.

In-process sensing and measurement will provide the capability for automatic feedback of control signals which is required for future analog and/or computer control systems at RAAP and other facilities.

#### RECOMMENDATIONS

Modifications to the developed prototype nitric acid analyzer,  $\text{HNO}_3/\text{H}_2\text{SO}_4$  analyzer, and the TNT set-point analyzer, as determined by the currently on-going evaluation and debugging program, should be implemented in a future program in order to optimize these control instrumental systems.

Other ammunition production processes, such as the continuous RDX-Composition B lines currently undergoing modernization, should be surveyed with respect to their requirements in regard to process-control instrumentation procurement and development.

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- OPEN-LOOP CONTROL
- MECHANICAL TIME SEQUENCING CONTROL
- PROGRAMMABLE DIGITAL CONTROL
- CLOSED-LOOP CONTROL
- AUTOMATIC ANALOG
- DIRECT DIGITAL COMPUTER CONTROL

Fig 1 Automatic manufacturing process-control categories



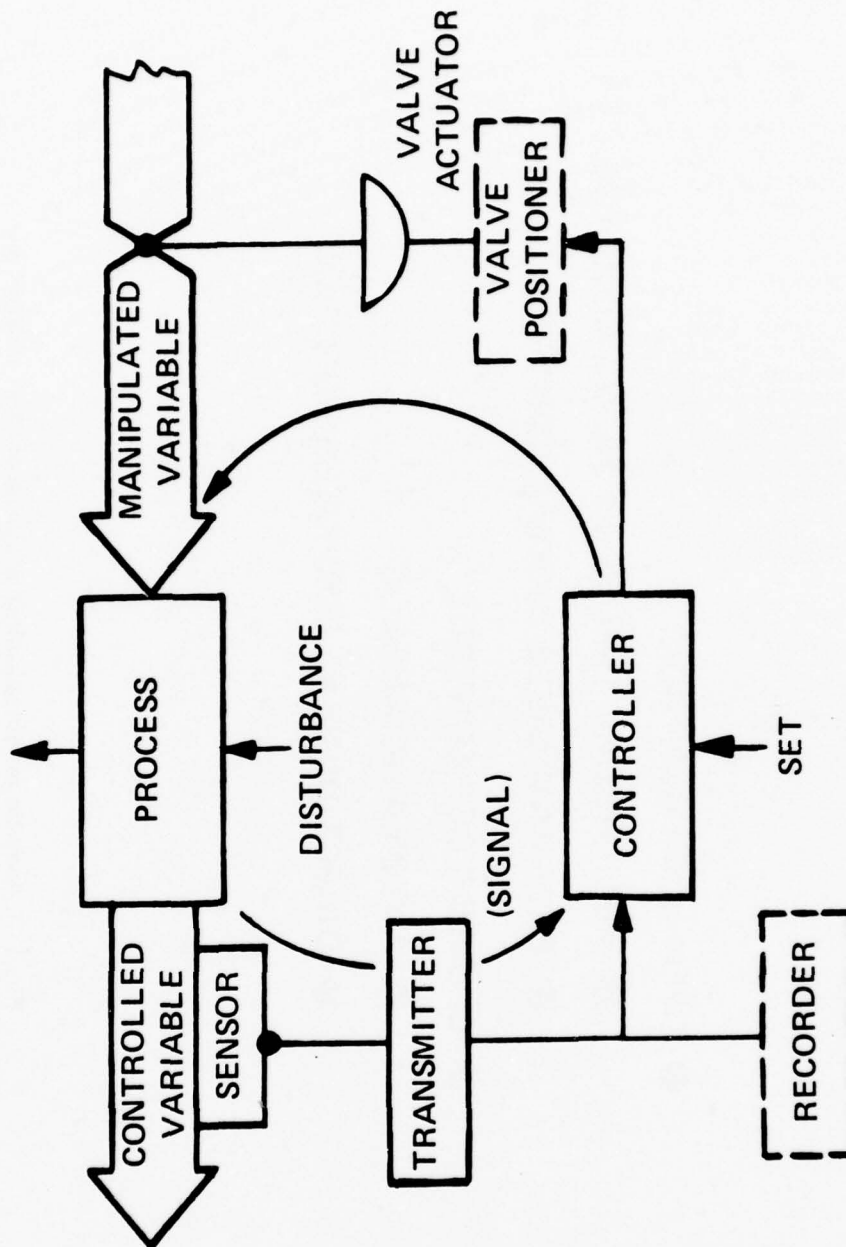


Fig 2 Closed-loop control

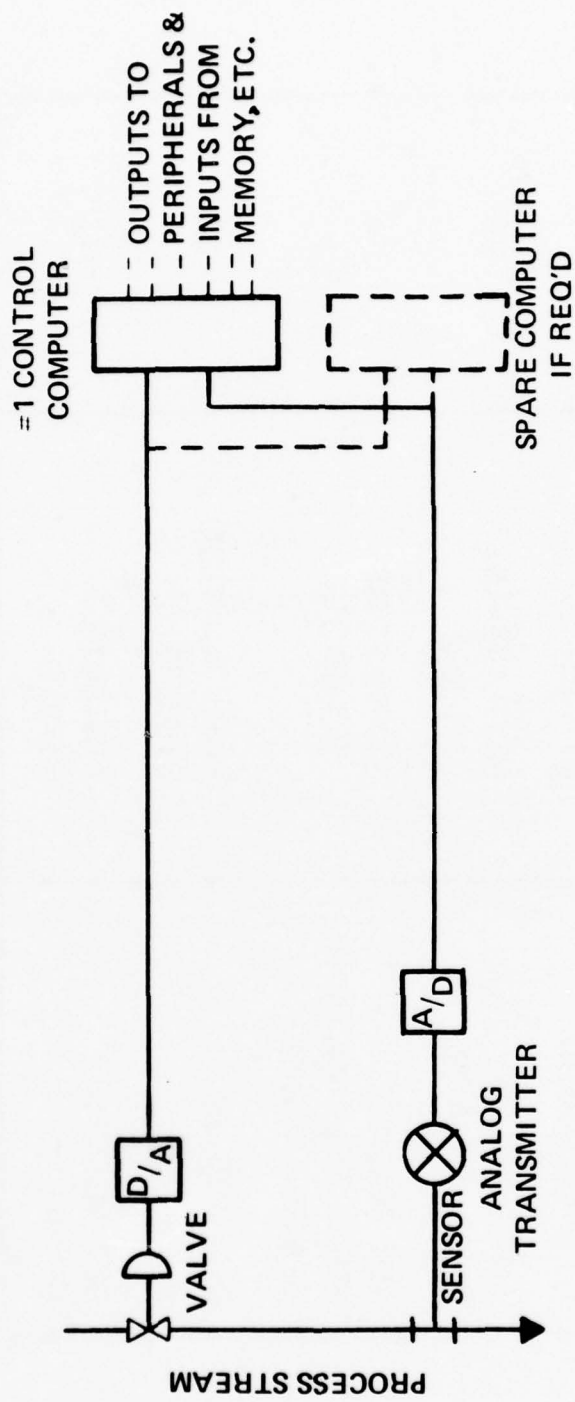


Fig 3 Over-simplified concept of direct digital control

MEASURED VARIABLE	LOCATION	NO. OF TESTS / DAY
NITRIC ACID IN SPENT ACID	ALL NITRATOR STAGES (8)	72
TOTAL ACID IN SPENT ACID	NITRATORS 1 & 2	18
SET POINTS (CRYSTALLIZATION TEMPERATURE)	NITRATORS 3 THRU 8	14
SET POINTS	PURIFICATION STAGES	24
SET POINTS (FINAL)	FINISHING STAGE	12

Fig 4 TNT process-control parameters

- CONTINUOUS TNT PROCESS
  - AUTOMATIC ON-LINE UV NITRIC ACID ANALYZER
  - AUTOMATIC ON-LINE HPLC NITRIC/SULFURIC ACID ANALYZER
  - AUTOMATIC ON-LINE TNT SET POINT SYSTEM
- AUTOMATED HIGH EXPLOSIVES MELT POUR LINE
  - EXPLOSIVE PUMP MALFUNCTION DETECTION AND CONTROL SYSTEM

Fig 5 Developed instrumentation for process control in manufacture of energetic materials



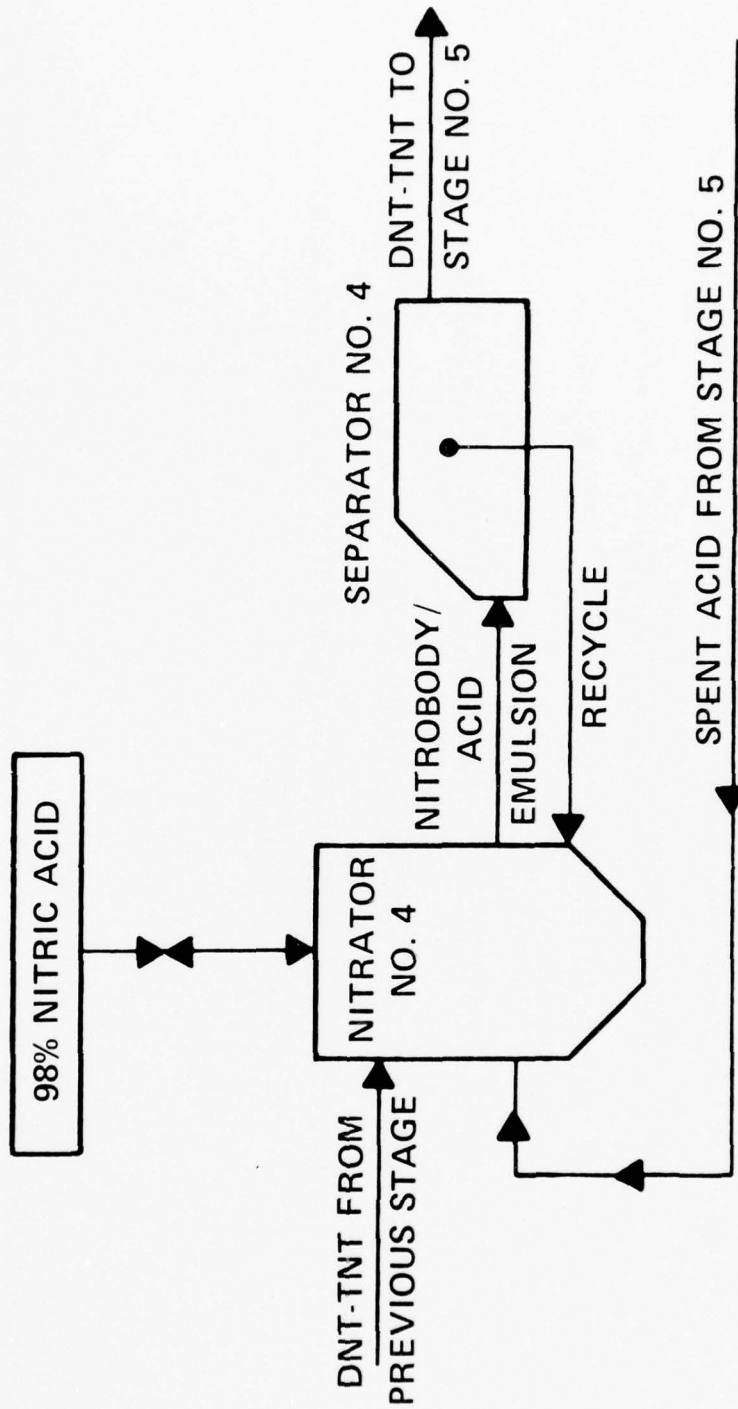


Fig 6 Typical nitration stage

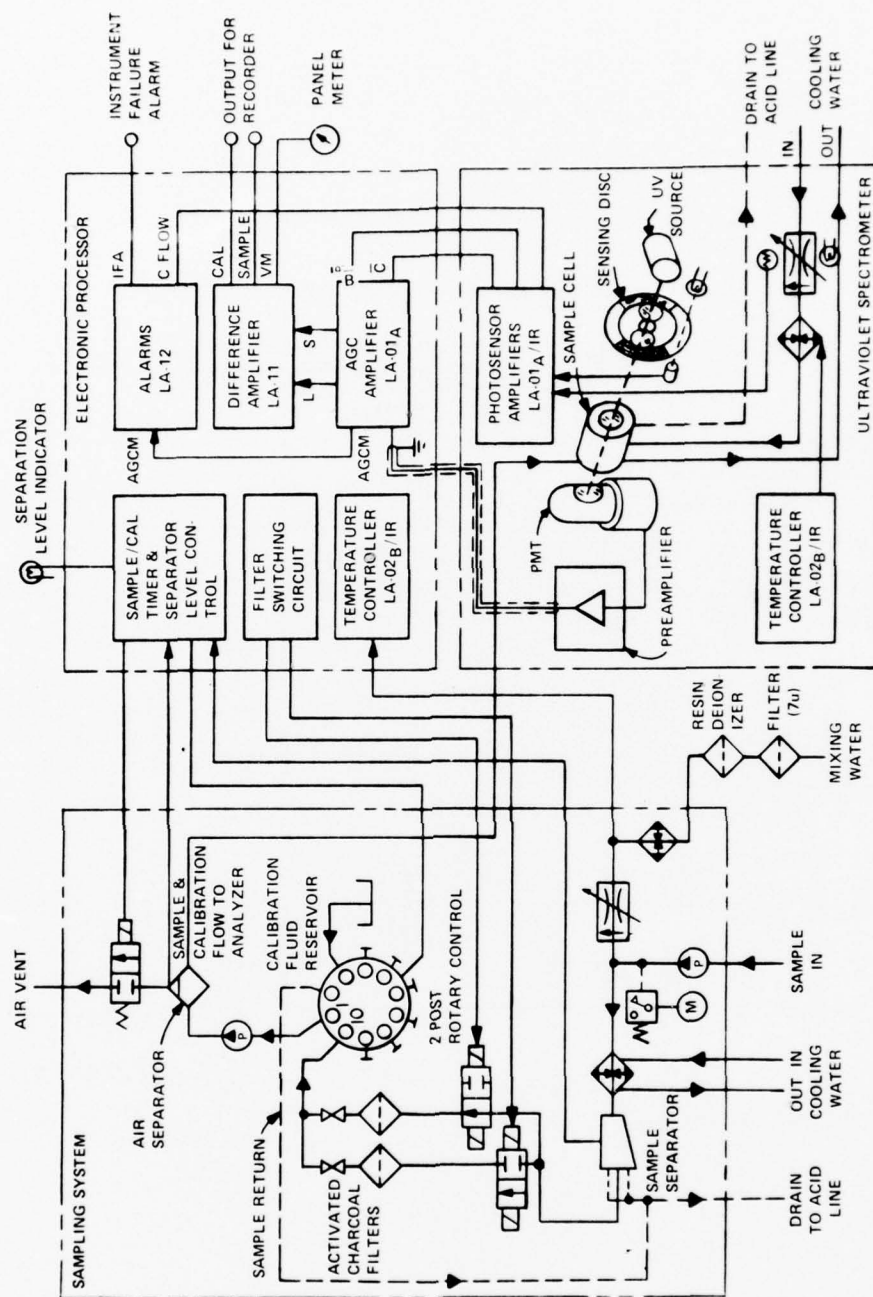


Fig 7 Nitric acid analyzer system

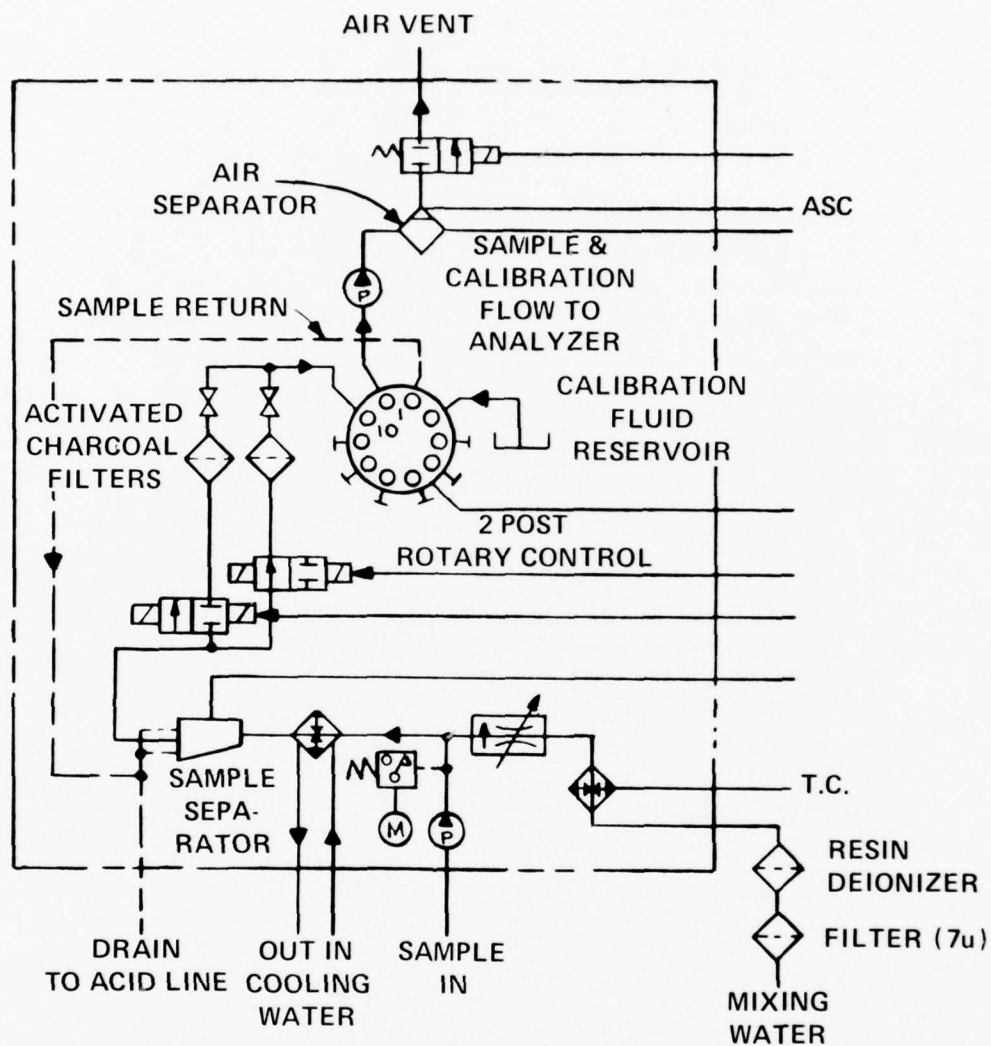


Fig 8 Sampling System

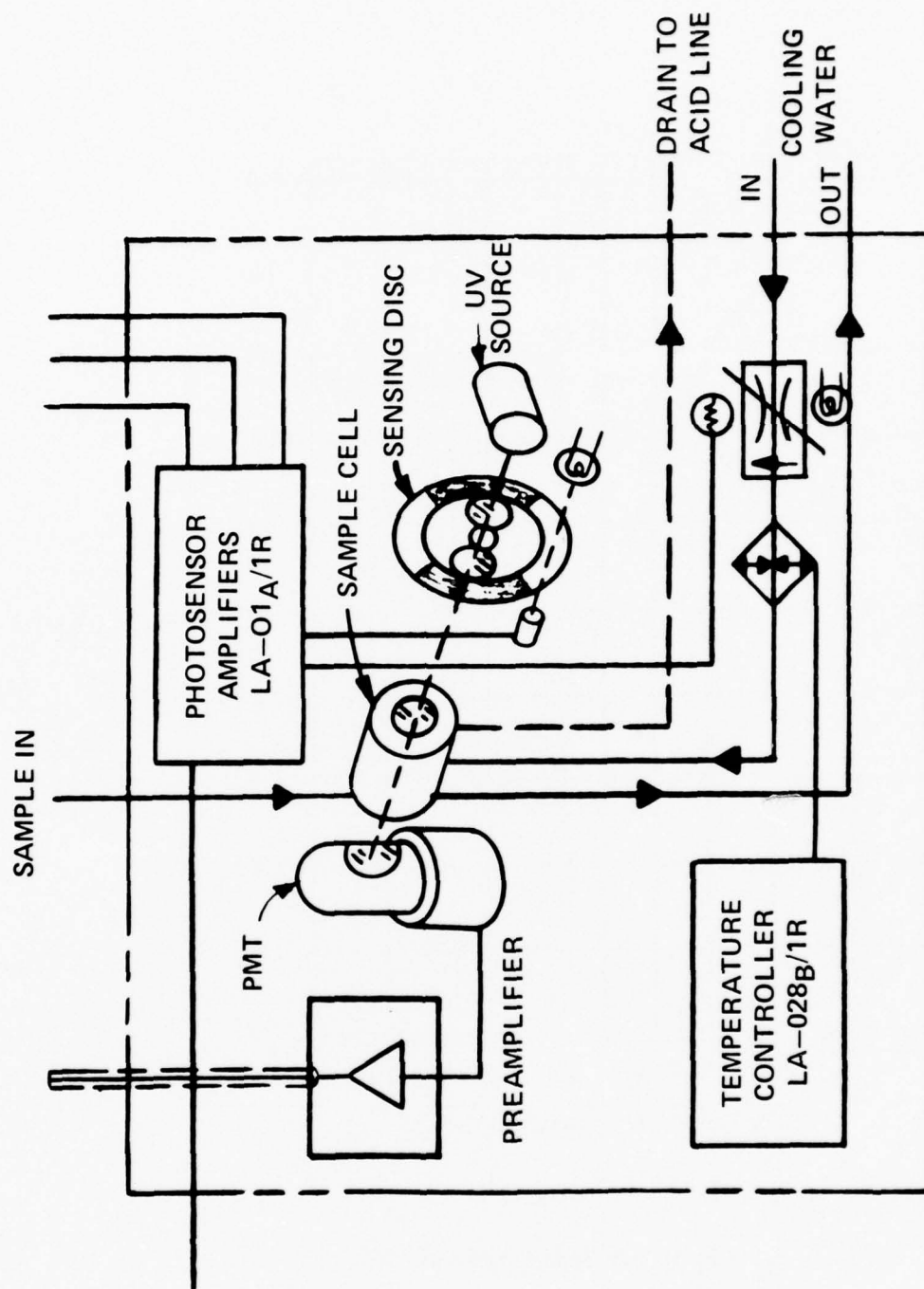


Fig 9 Ultraviolet spectrometer



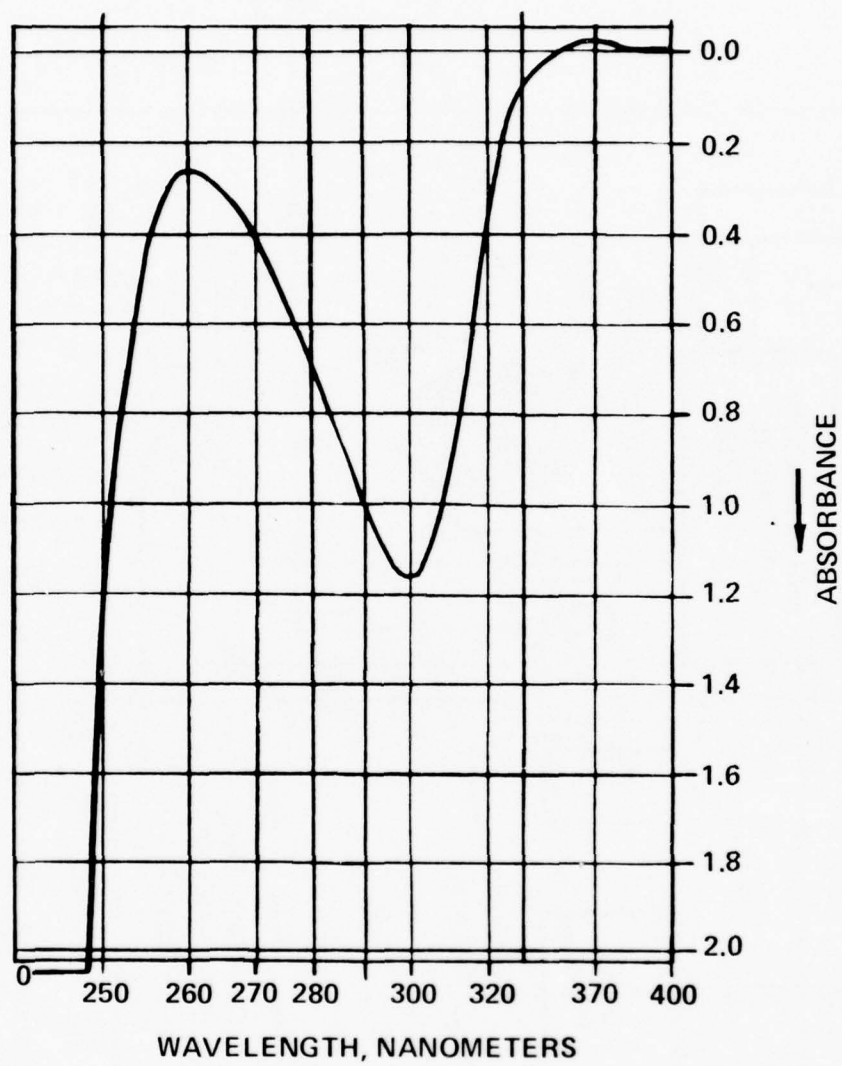


Fig 10 UV absorbance of  $\text{HNO}_3$

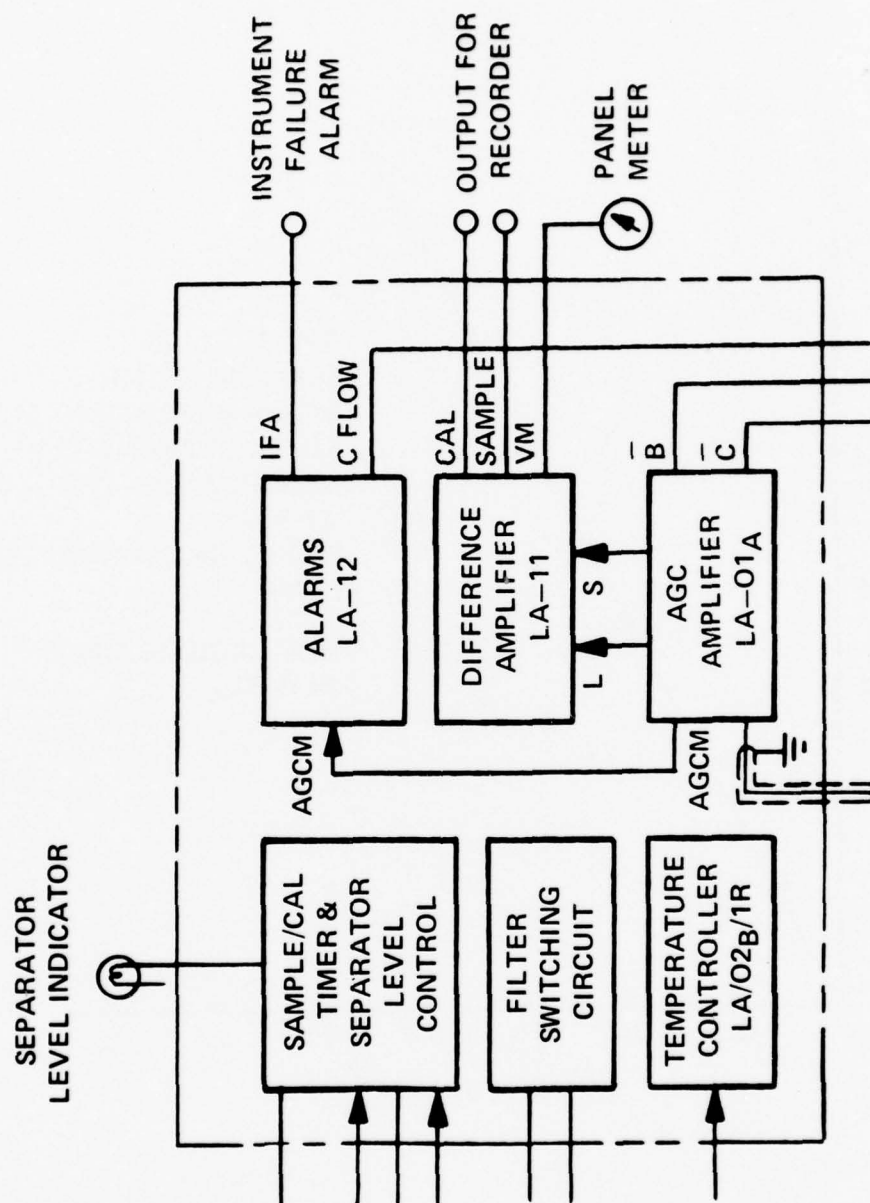


Fig 11 Electronic processor

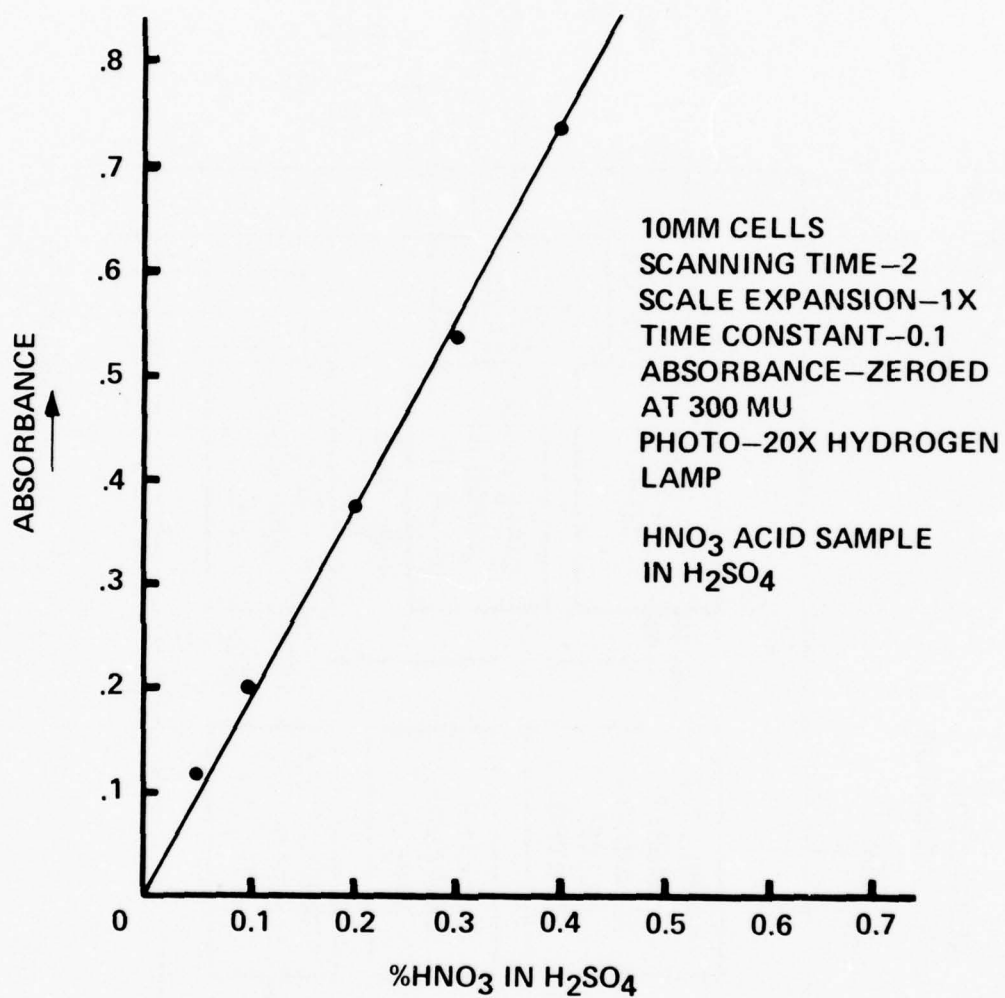


Fig 12 Absorbance of HNO<sub>3</sub> as a function of concentration

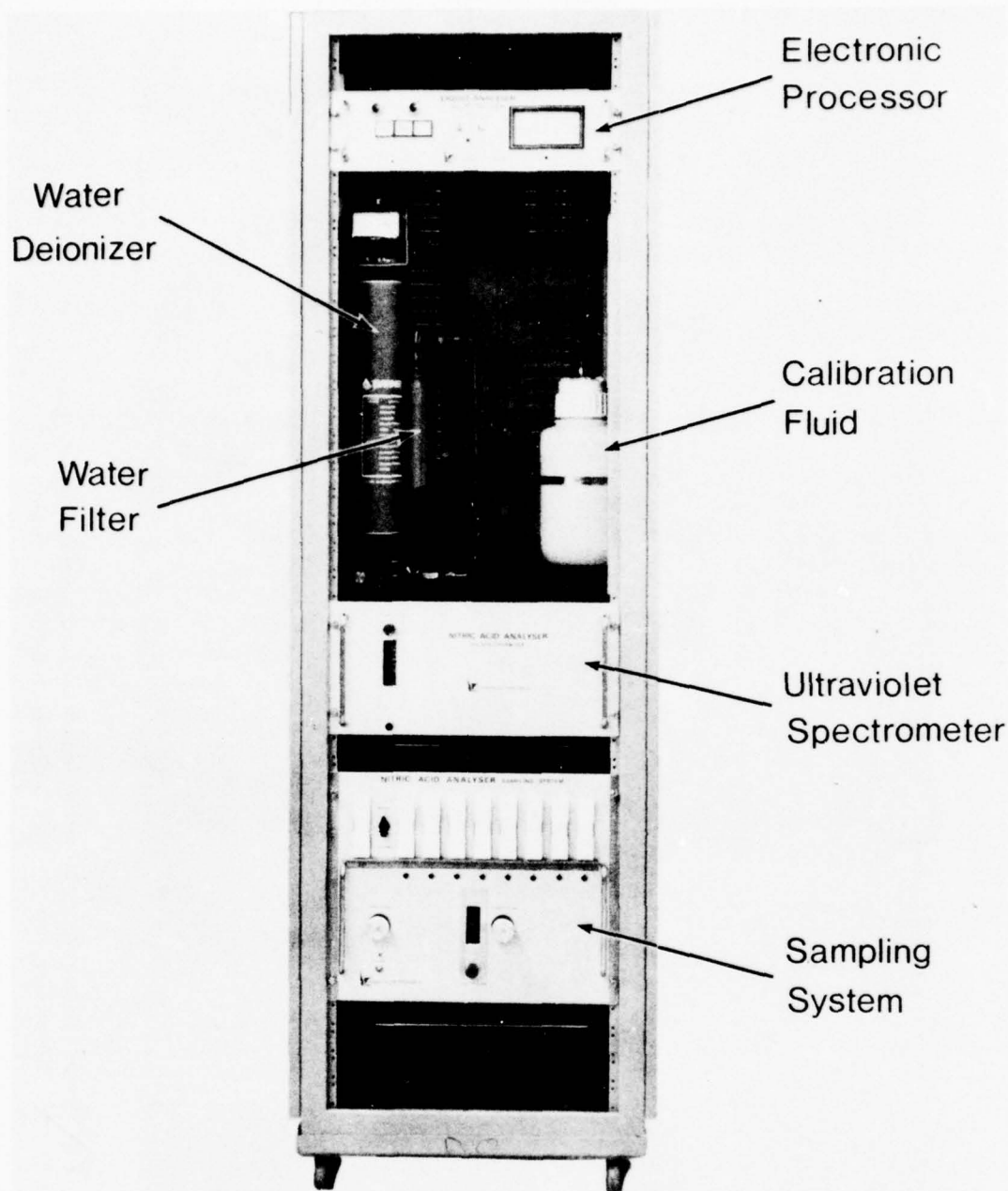


Fig 13 Nitric acid analyzer



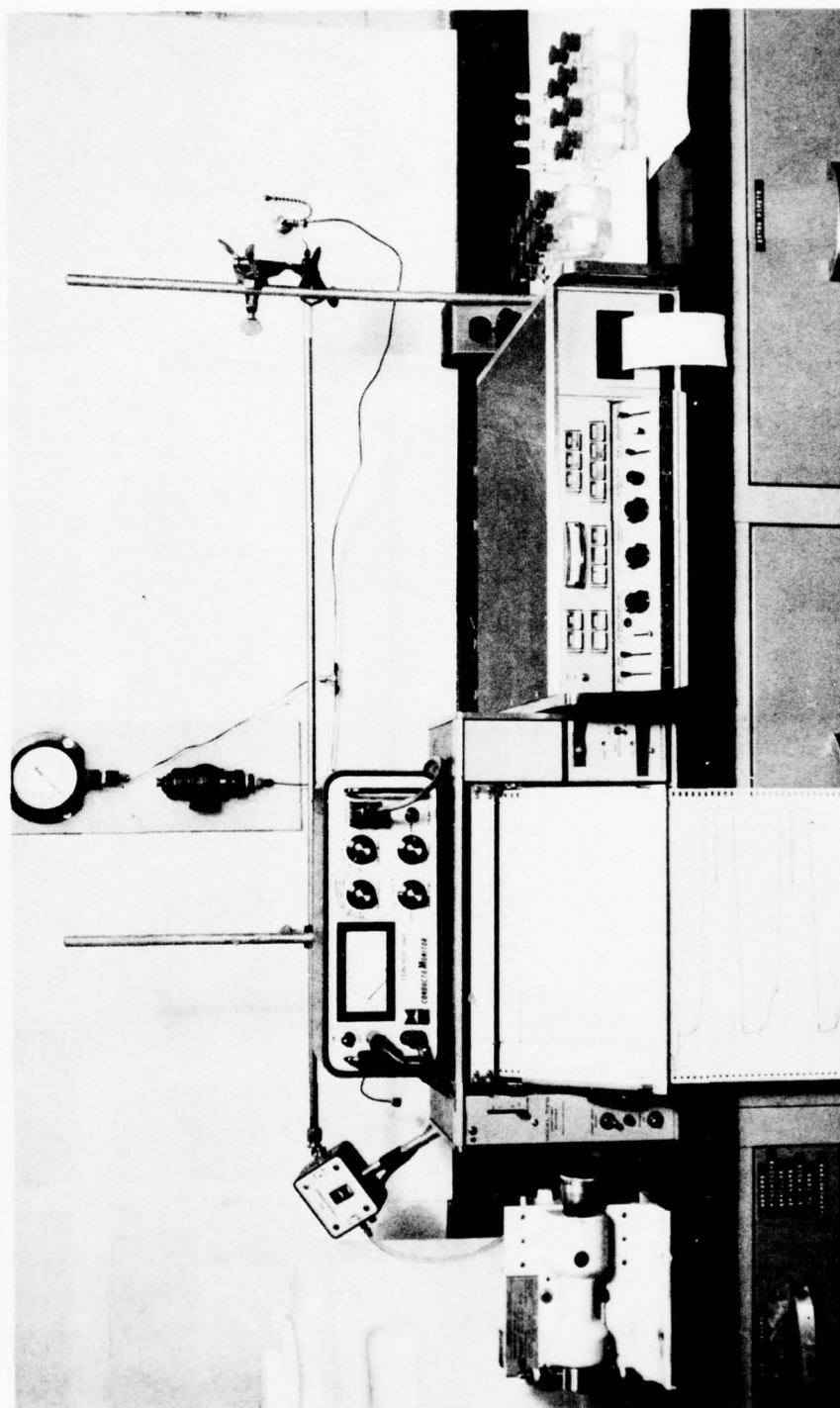


Fig 14 Breadboard installation of chromatic system with signal amplifier/conditioner and electronic integrator

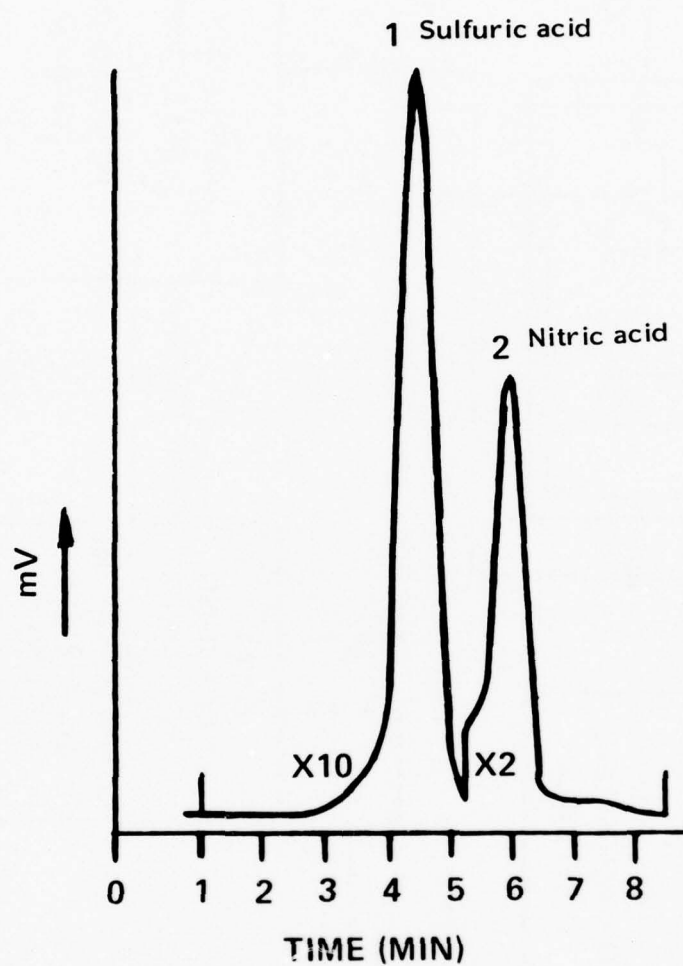


Fig 15 LC separation of sulfuric and nitric acids



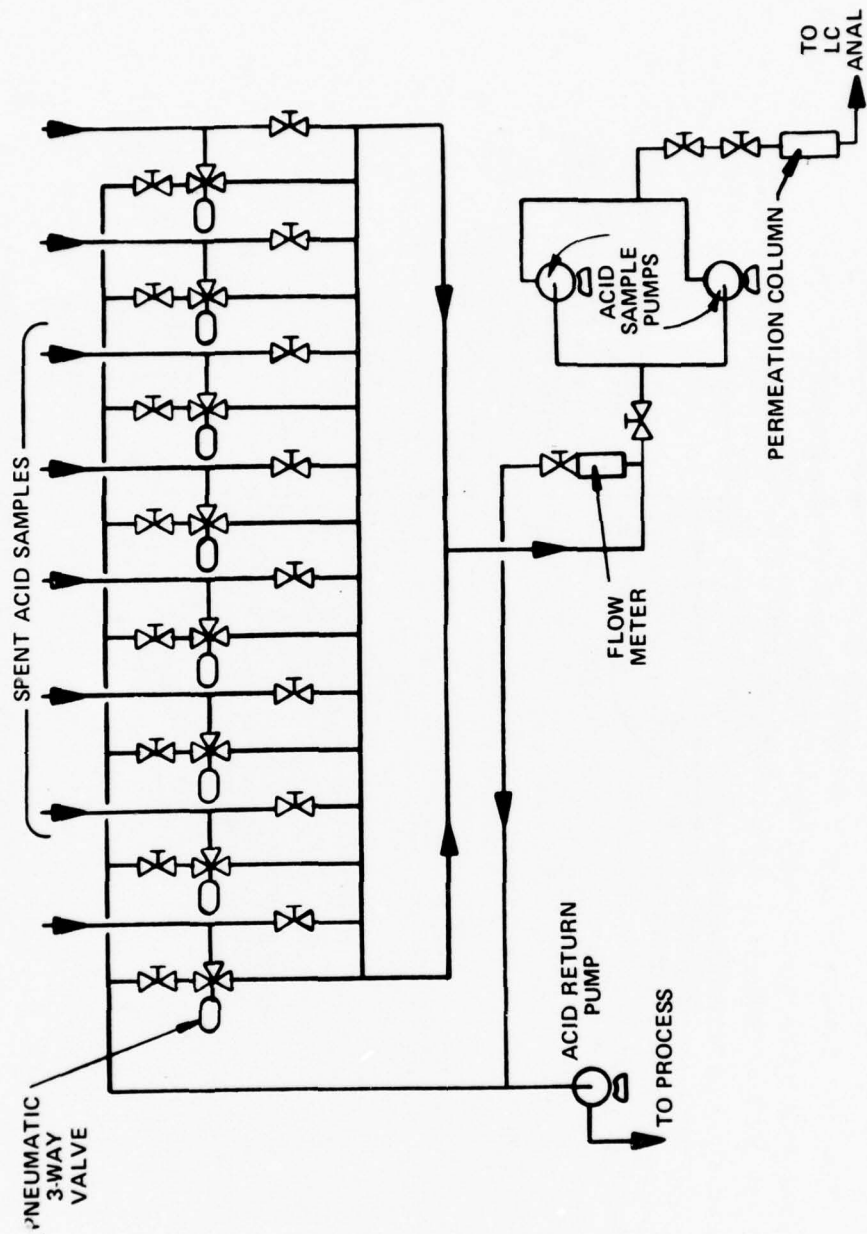


Fig 17 Acid stream selection and sample preparation for on-line liquid chromatographic acid analysis system



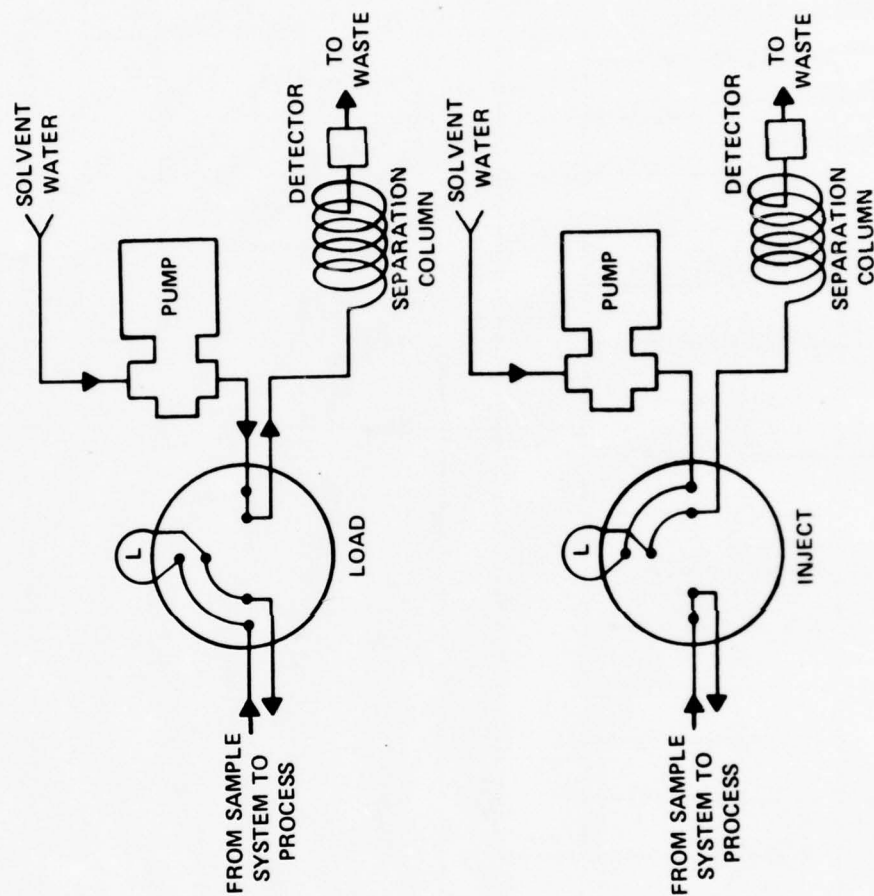


Fig 18 Schematic of acid-sample valve operation

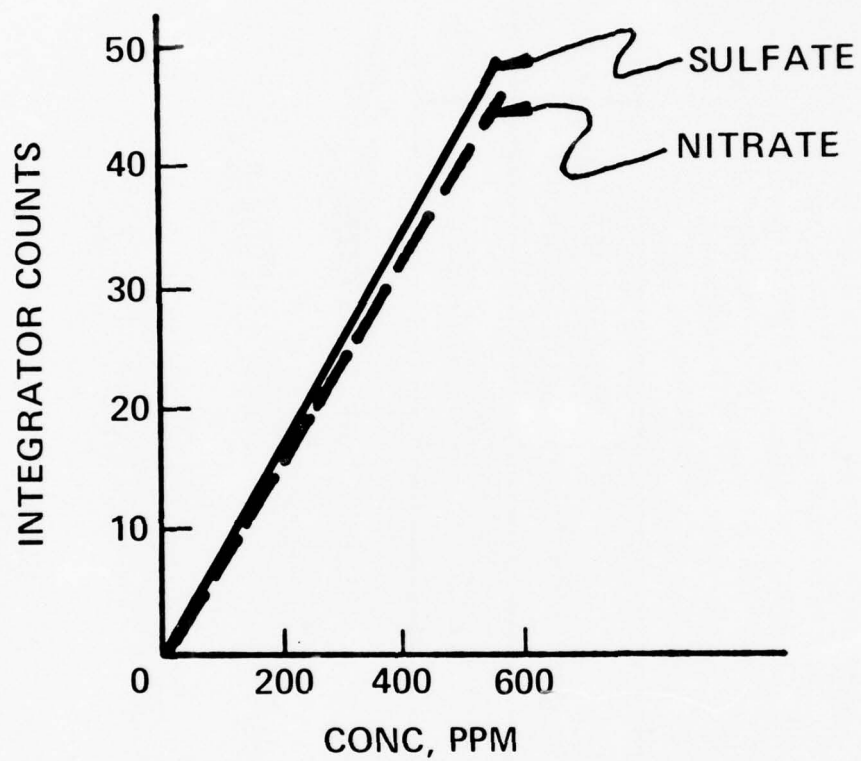


Fig 19 Linearity of sulfate-nitrate determination by LC

	INSTRUMENTAL	INSTRUMENTAL PLUS SAMPLING	FUNCTION OF TIME 5 MIN INCREMENTS
COEF OF VARIATION	0.25%	1.58%	2.30%

PRESENT METHOD 4.68%

Fig 20 N/S variability study

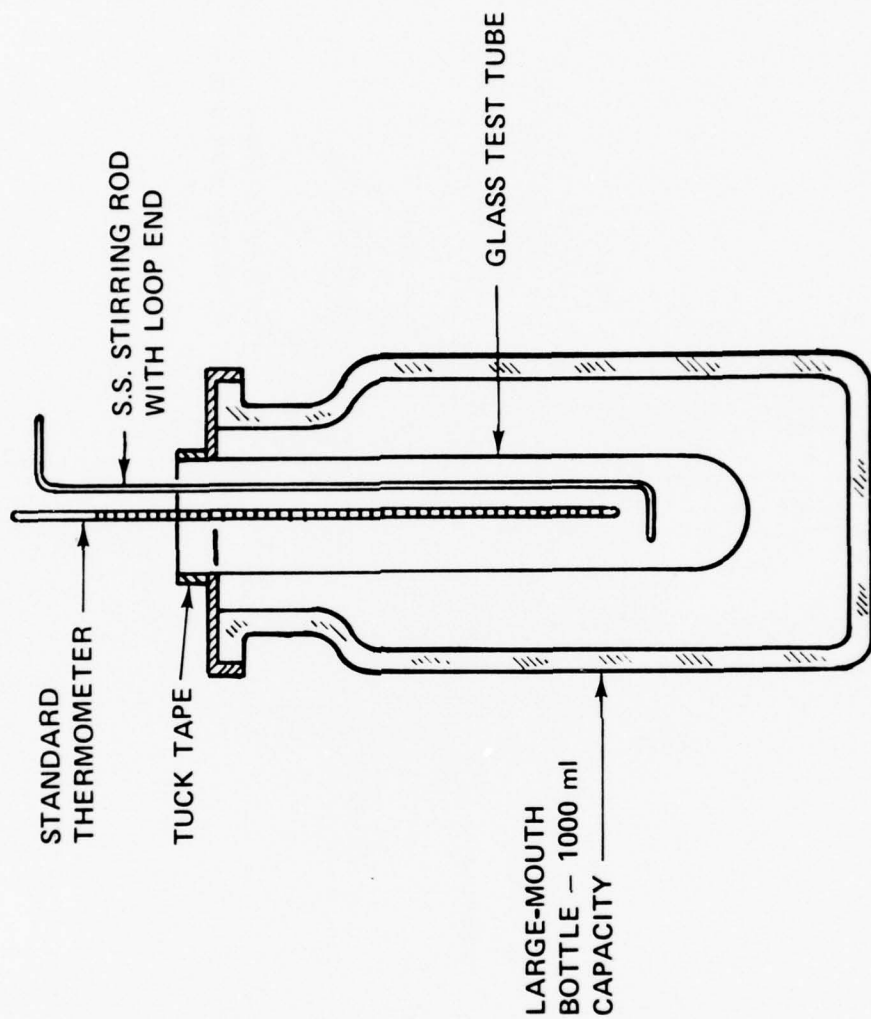


Fig 21 Standard solidification point apparatus

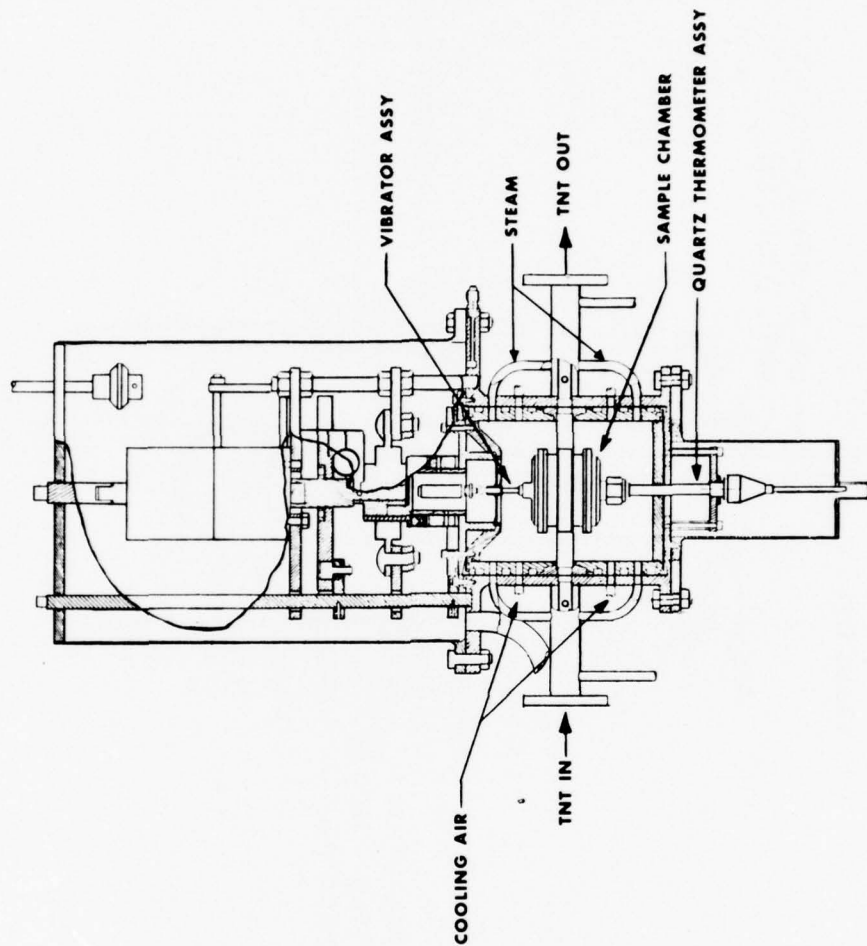


Fig 22 Automatic TNT set-point monitor



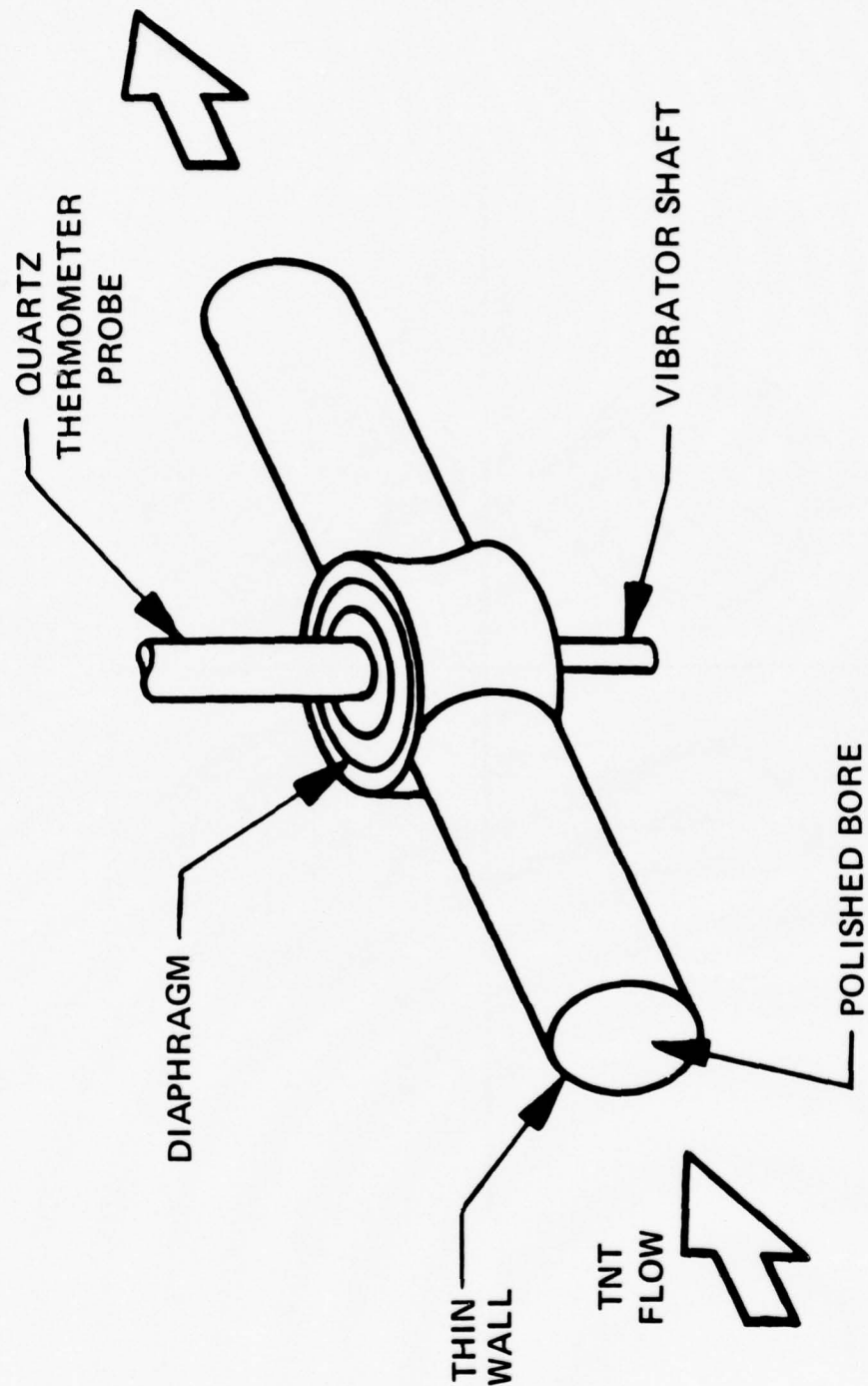


Fig 23 Roto-sampler test chamber

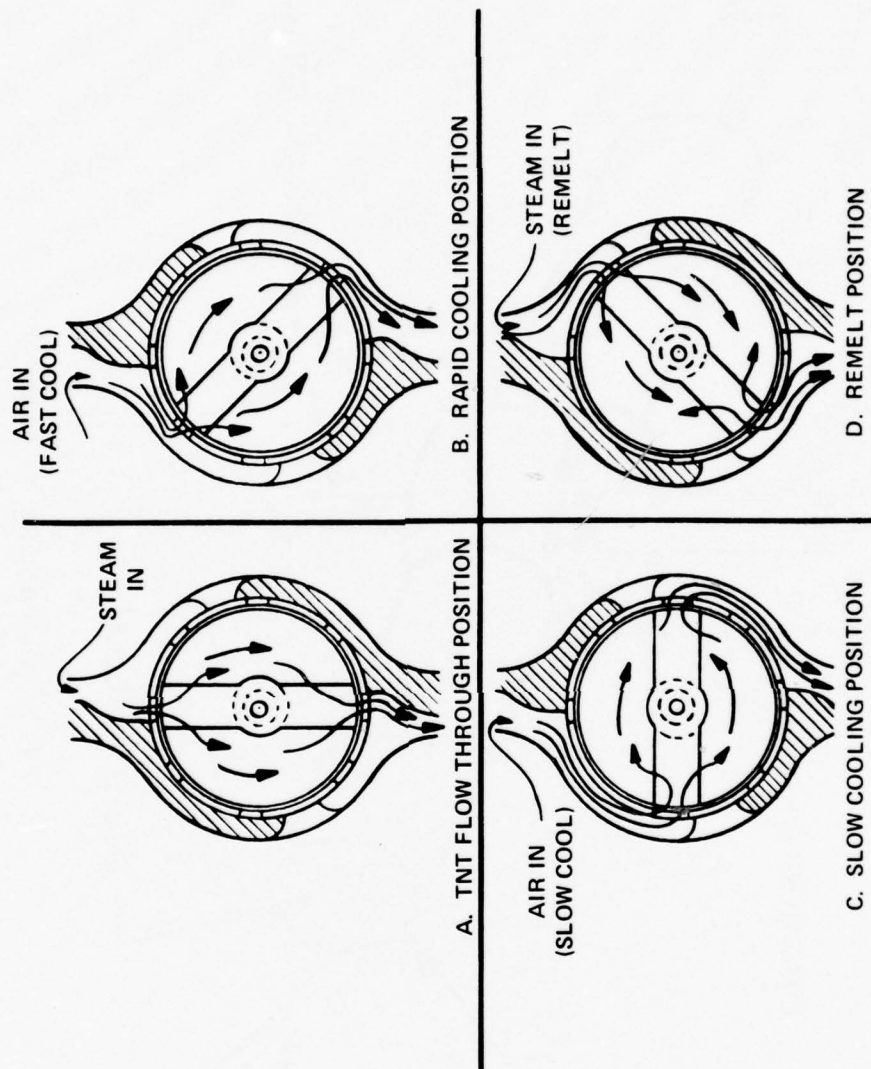


Fig 24 Roto-sampler during set-point determination cycle

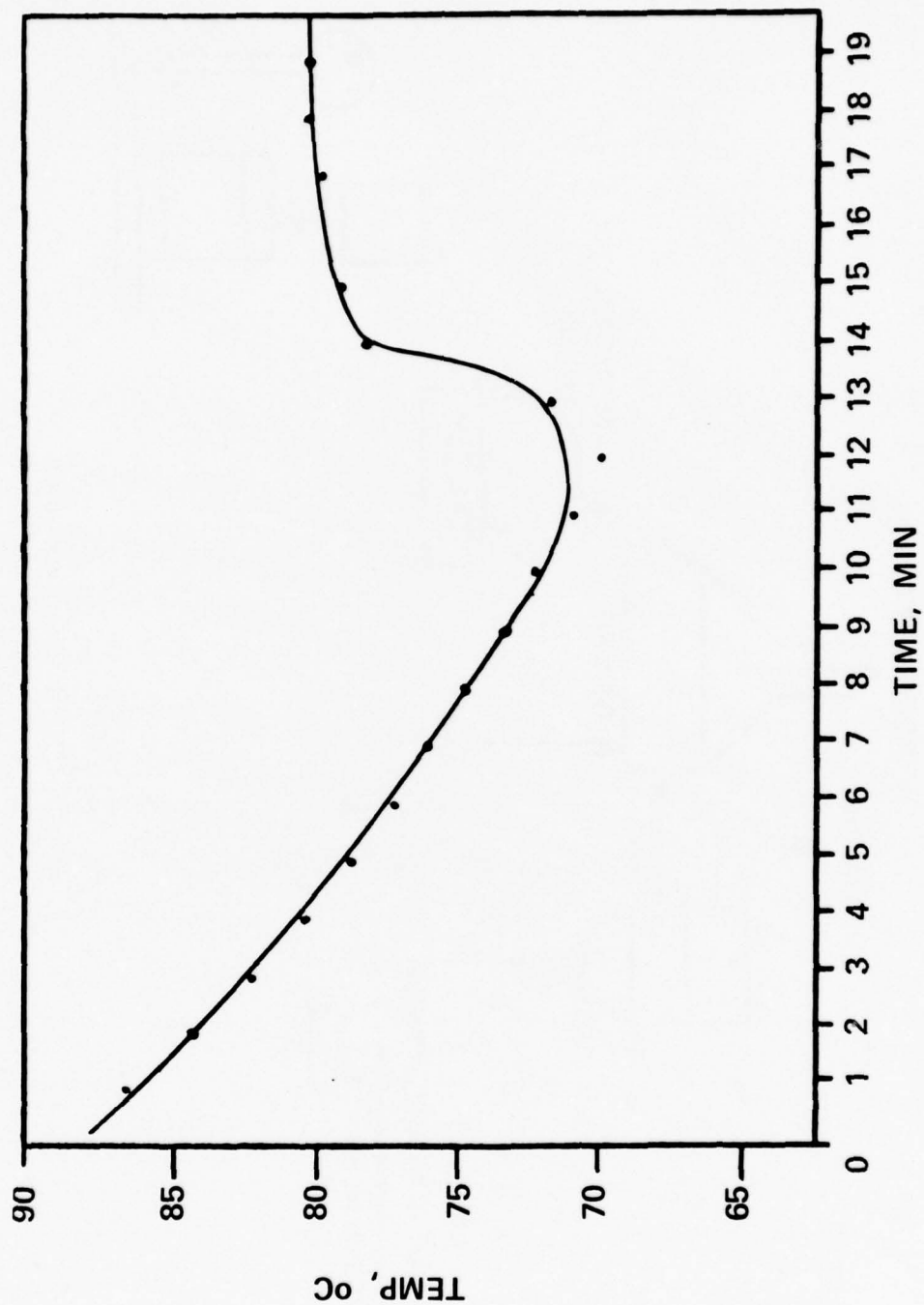


Fig 25 Cooling curve for molten TNT standard method

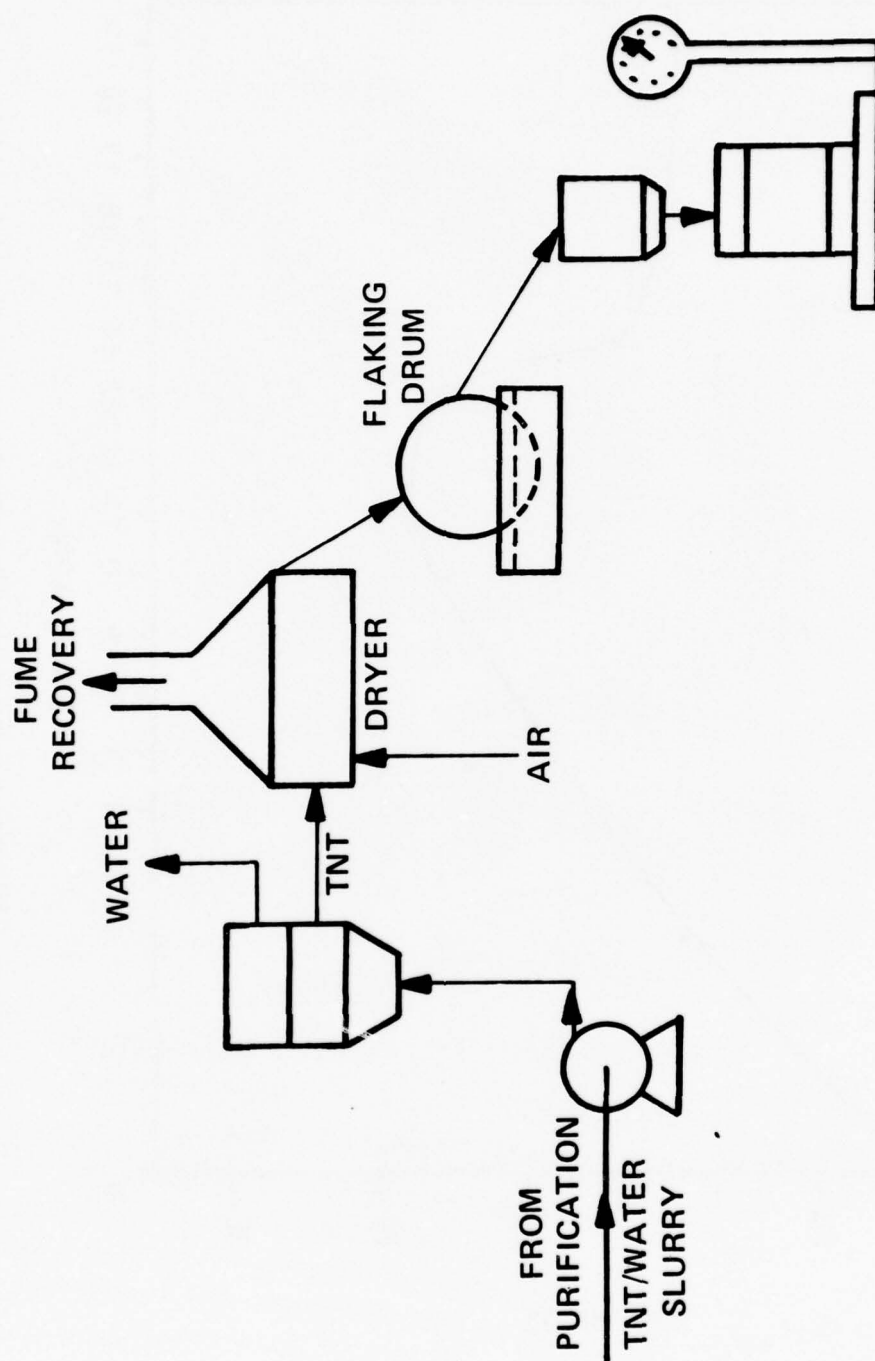


Fig 26 TNT finishing operation

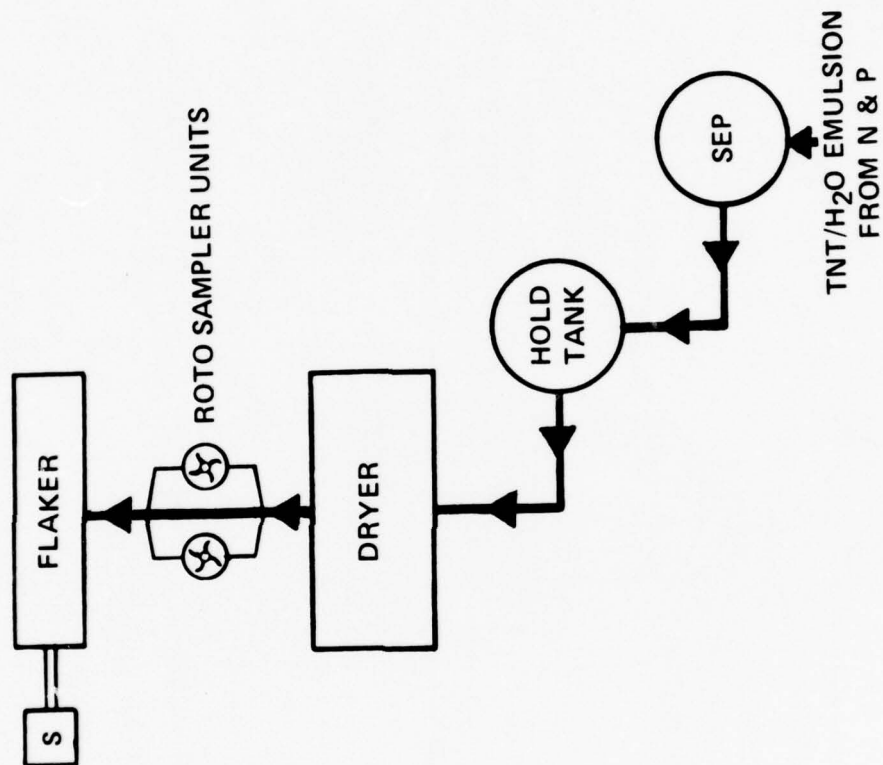


Fig 27 Location of roto-samplers in TNT finishing process



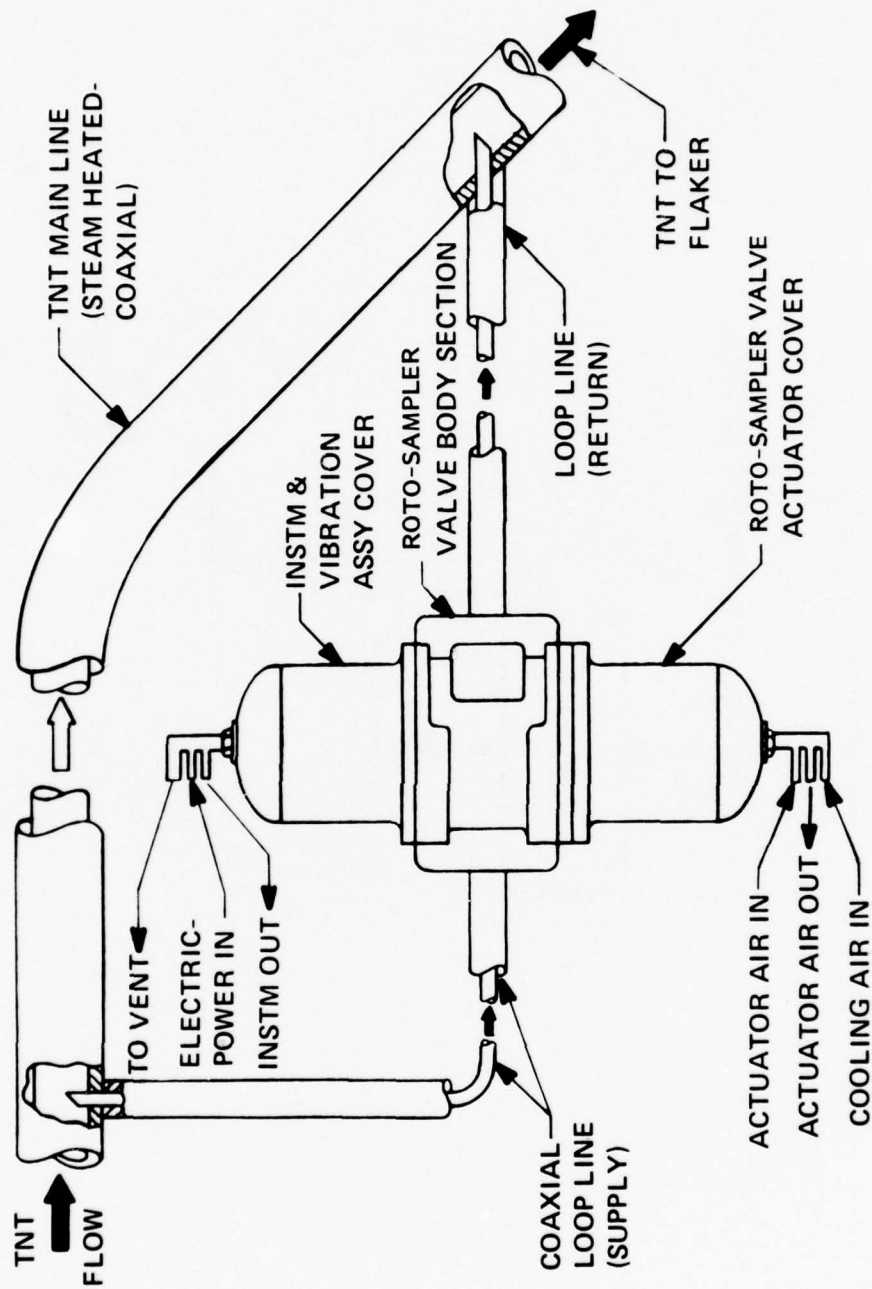


Fig 28 TNT set-point sampler

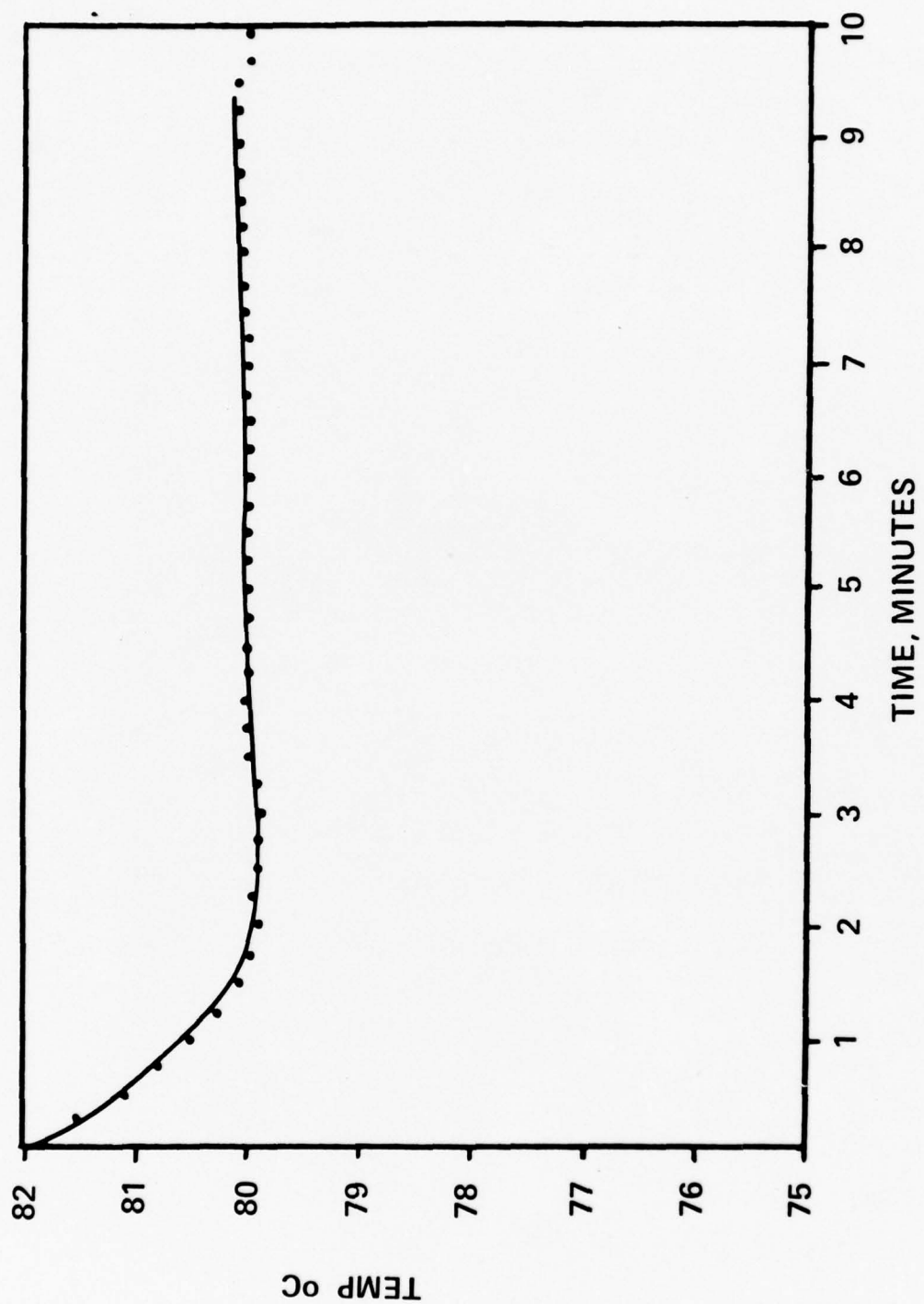


Fig 29 TNT vibrated externally

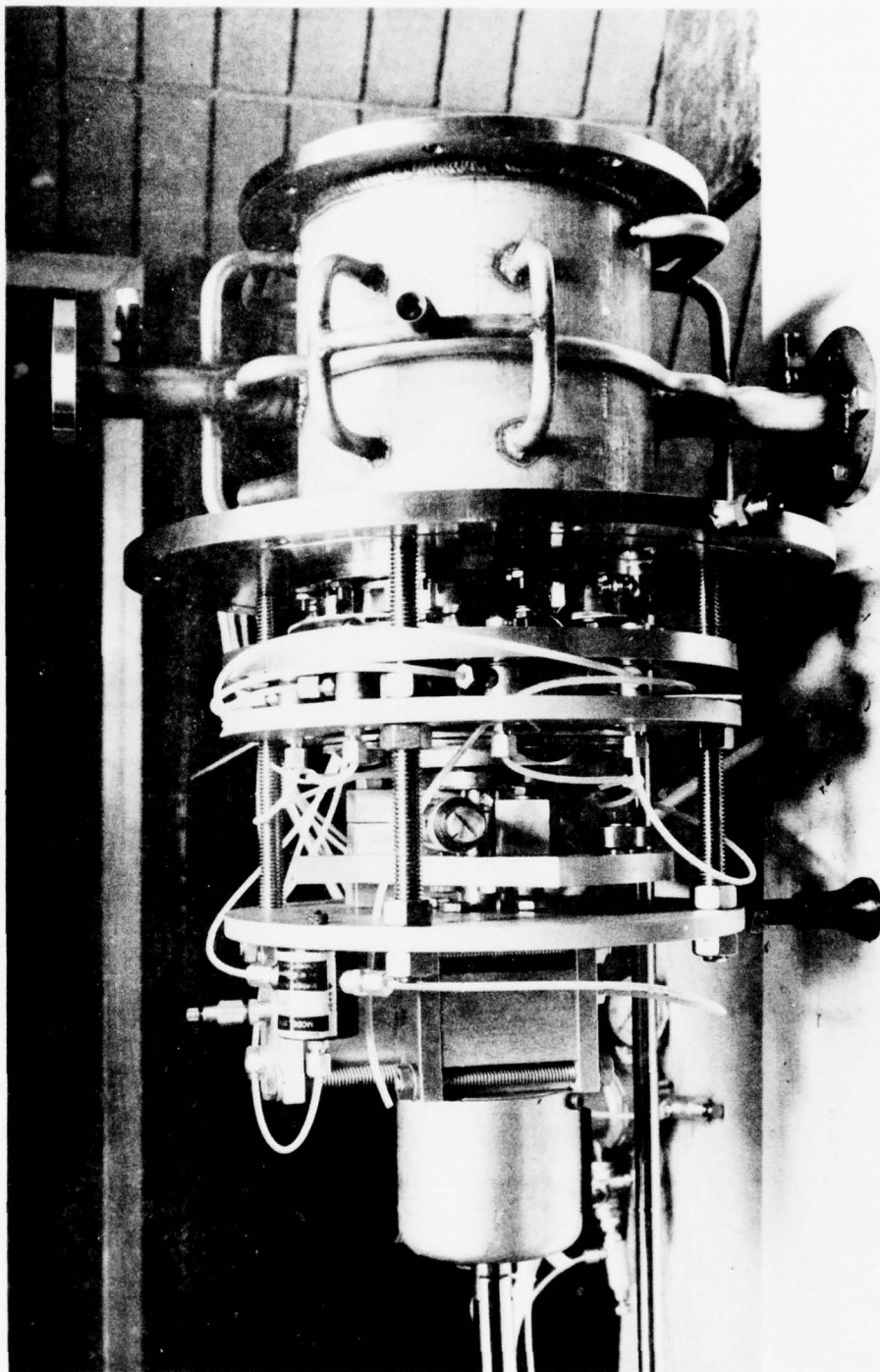


Fig 30 Roto-sampler



Fig 31 Computer-control system for roto-sampler

- ESTIMATED LABOR COST SAVINGS OF \$95,000 PER ANNUM – PER TNT LINE
- INCREASED PRODUCTION EFFICIENCY AS A RESULT IN REDUCED NEED FOR REWORKING OF LOW GRADE TNT
- OPTIMIZATION OF THE TNT PRODUCTION PROCESS THROUGH COMPLETE AUTOMATIC CONTROL
- ELIMINATE PRODUCTION LABORATORY IN N & P BUILDING (ONE PER TNT LINE)
- ENHANCED SAFETY OF THE TNT OPERATION SINCE FEWER PERSONNEL WILL BE REQUIRED

Fig 32 Benefits from on-line analyzer use in TNT process control



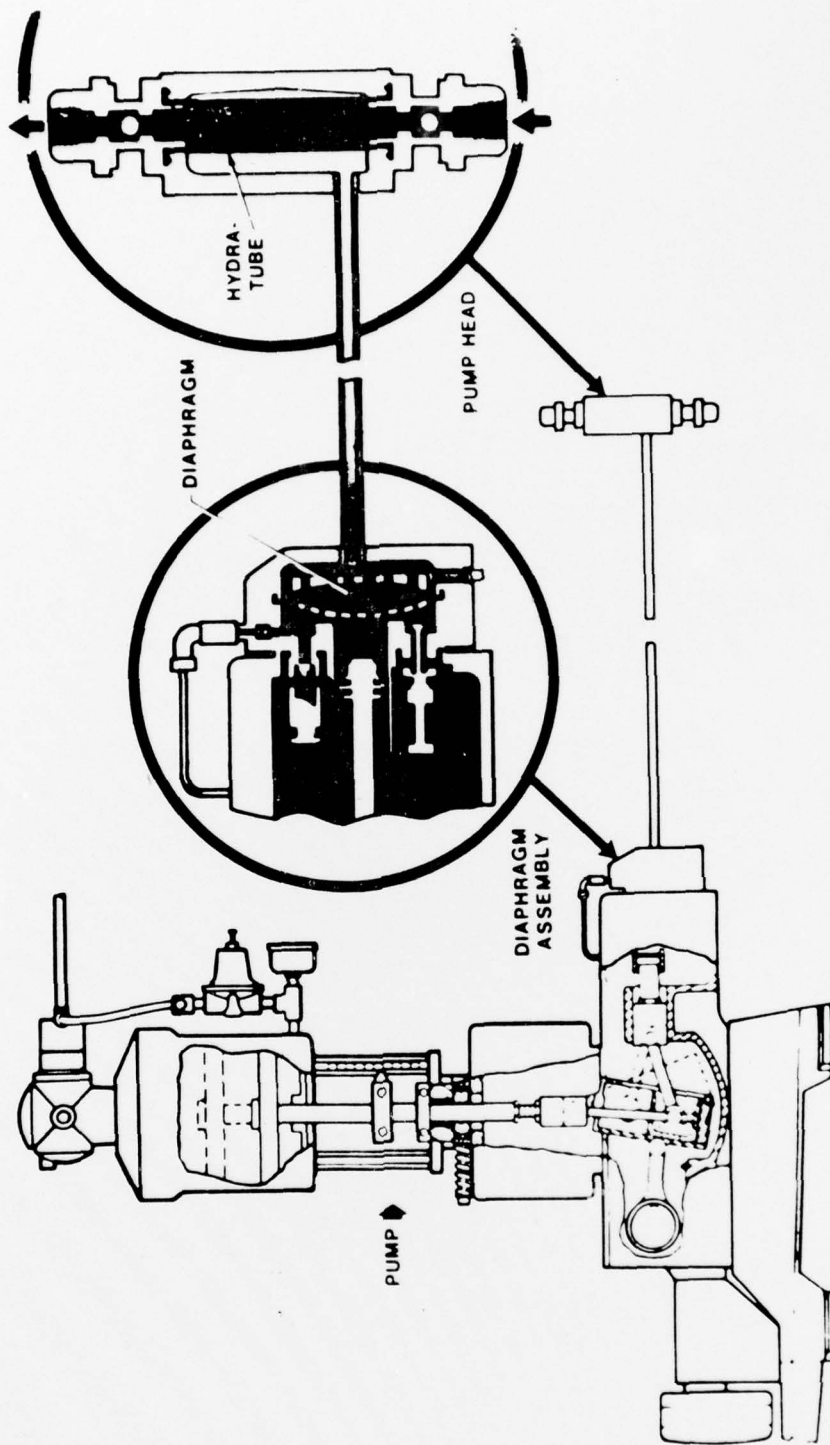


Fig 33 Pulsafeeder pump

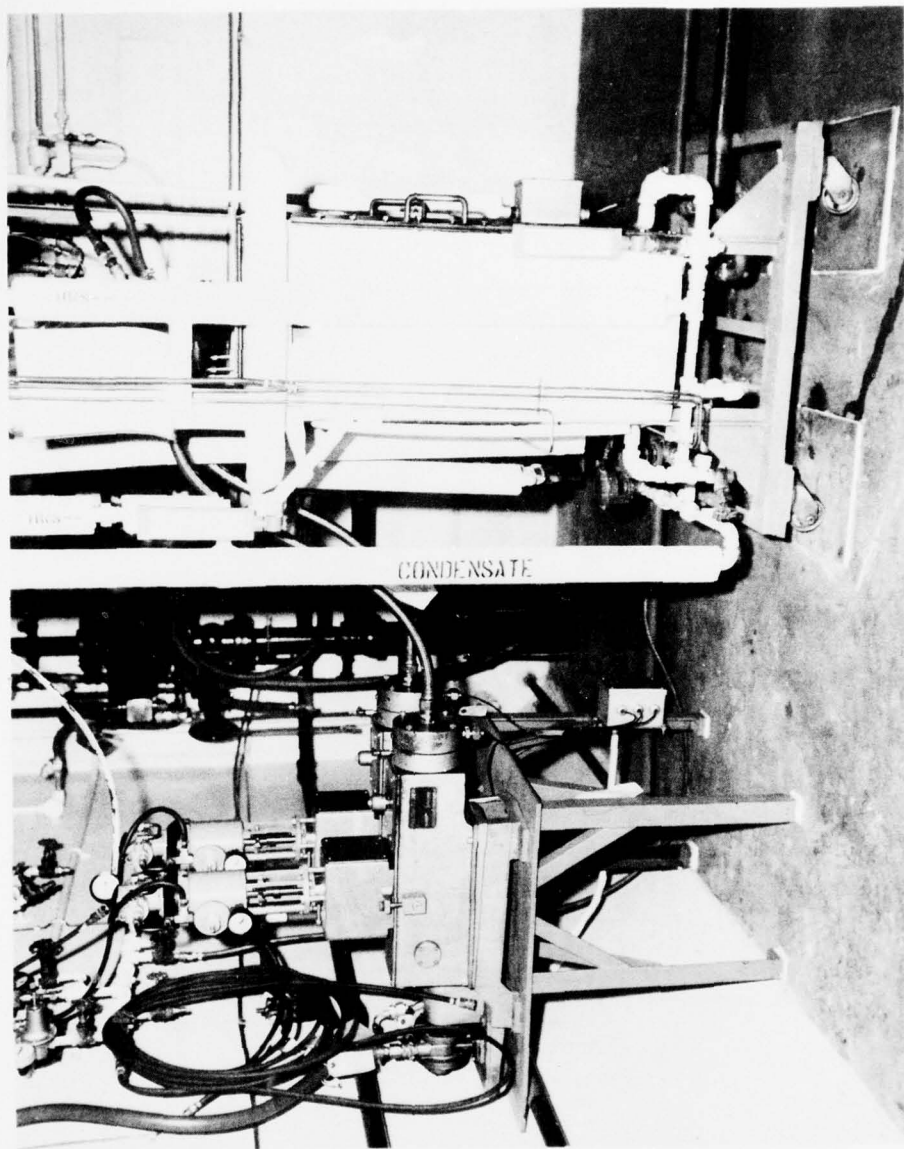


Fig 34 Pulsafeeder pump with pump heads in jacket

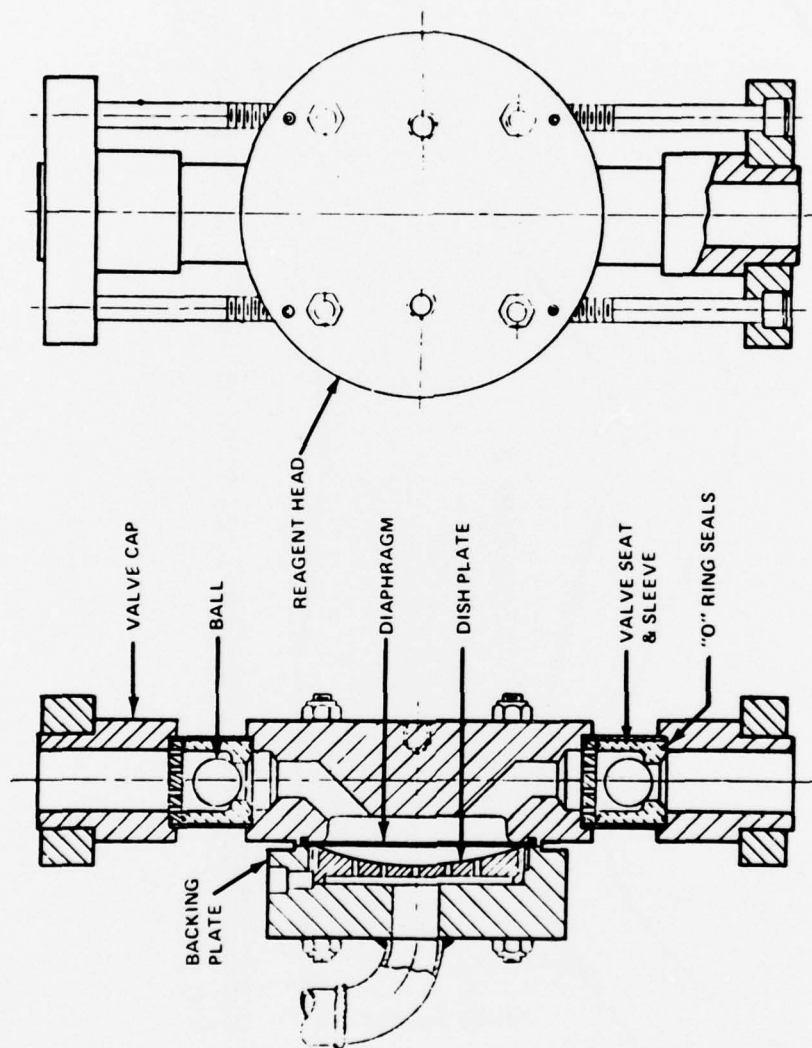


Fig 35 Duplex pulsafeeder remote head-diaphragm

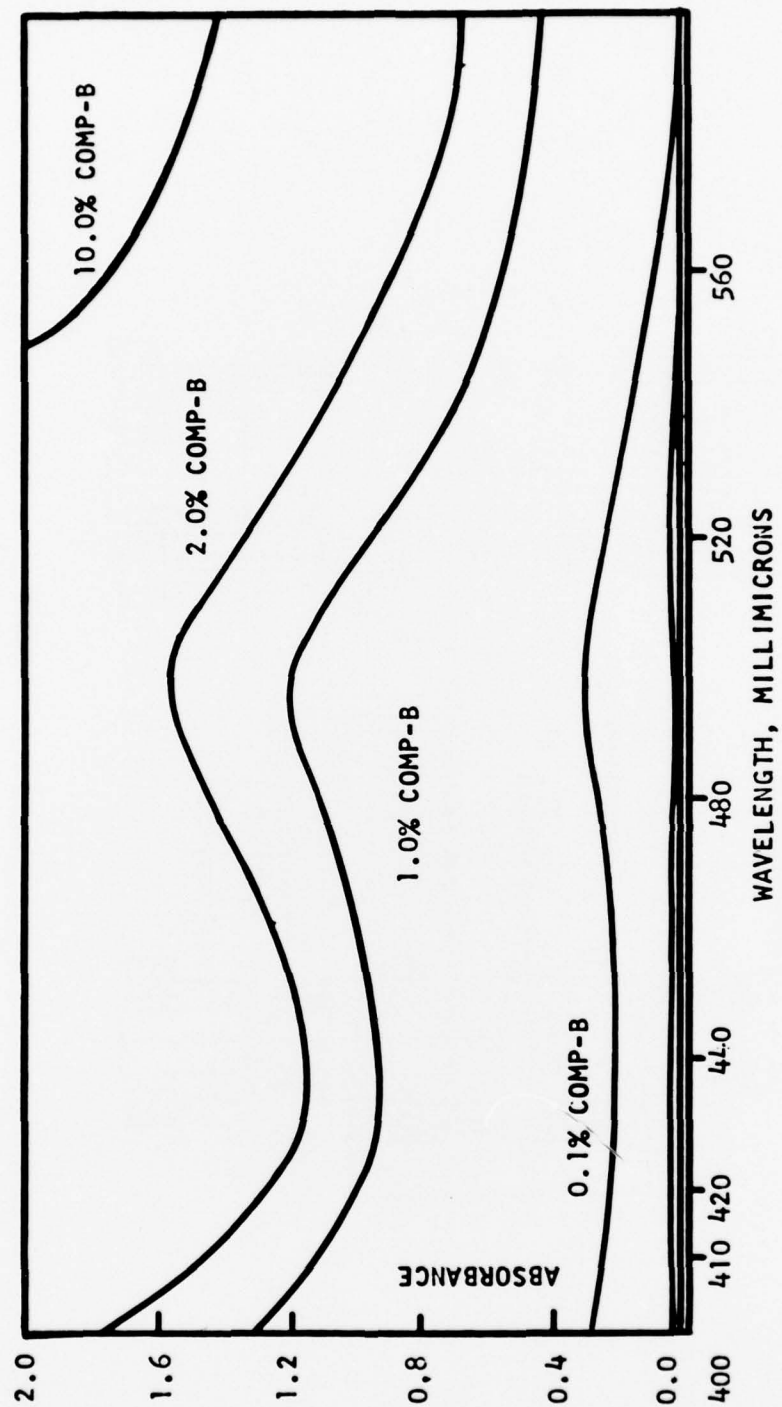


Fig 36 Hydraulic oil contaminated with composition B

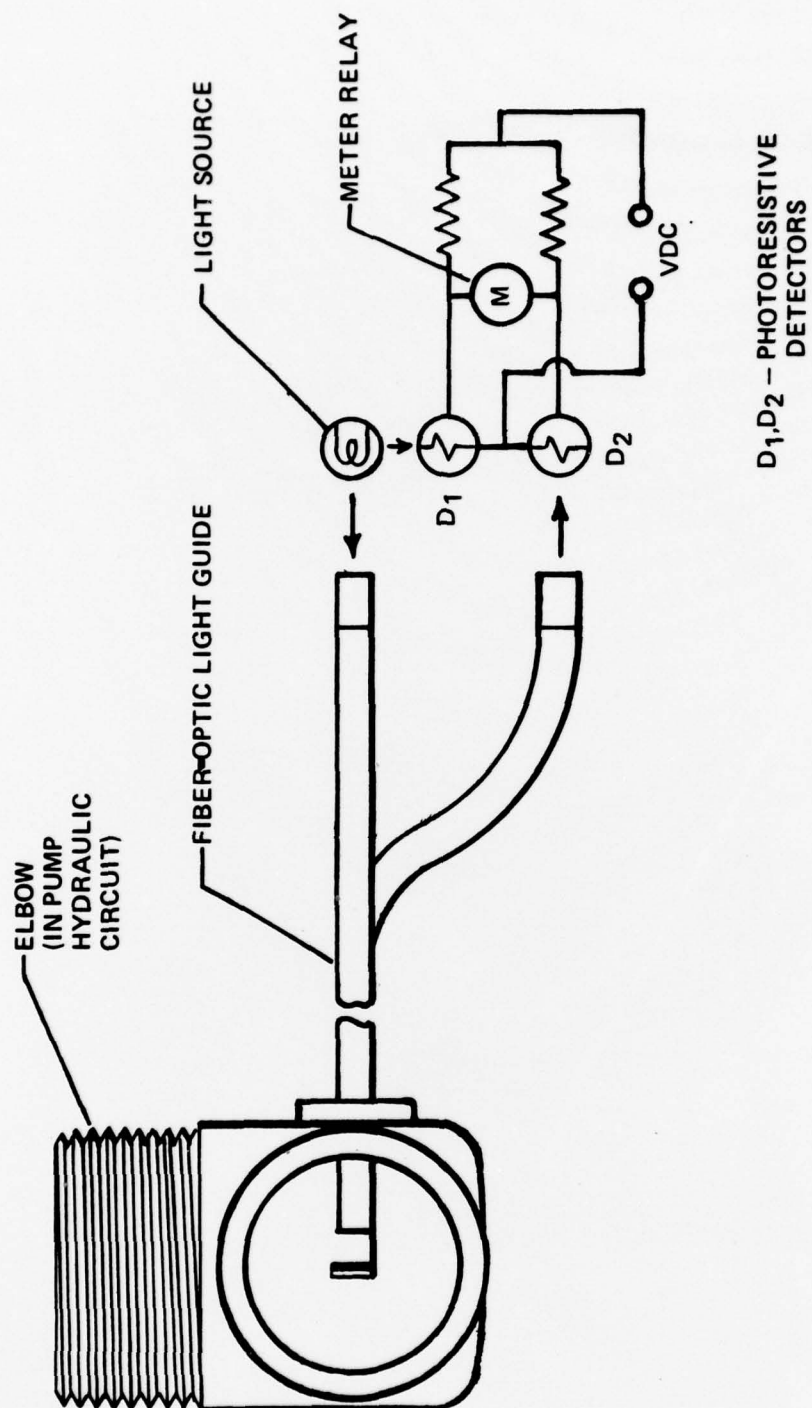


Fig 37 Pump diaphragm leak detector



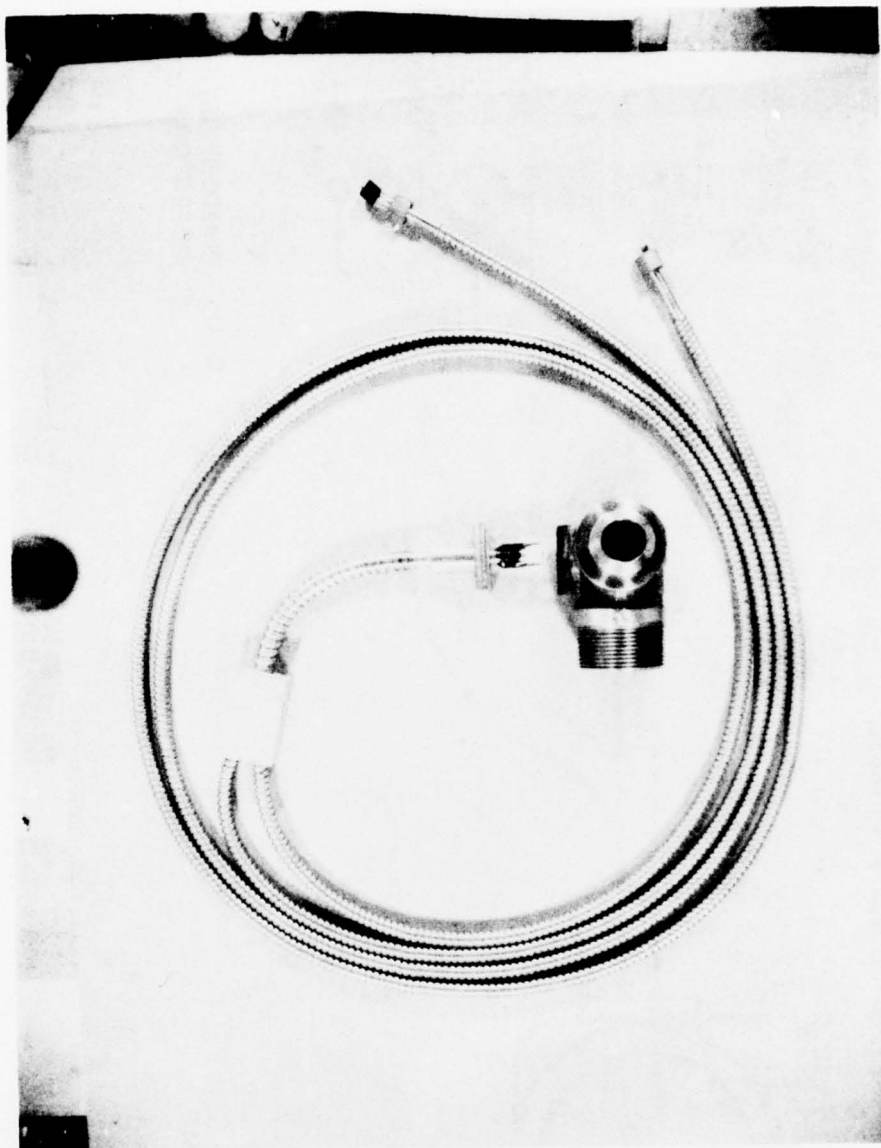


Fig 38 Bifurcated fiber-optic light guide with mounting elbow

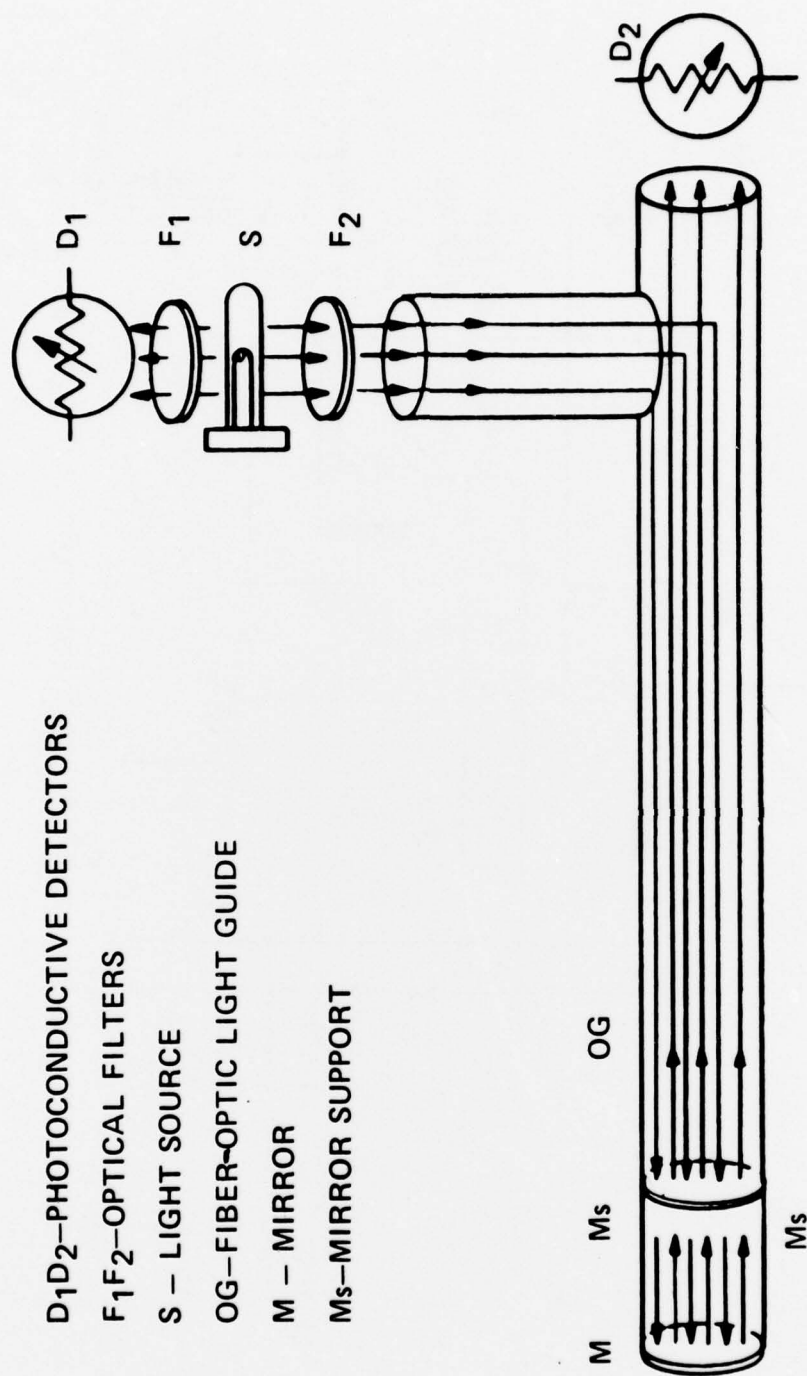


Fig 39 Schematic of instrument optical path

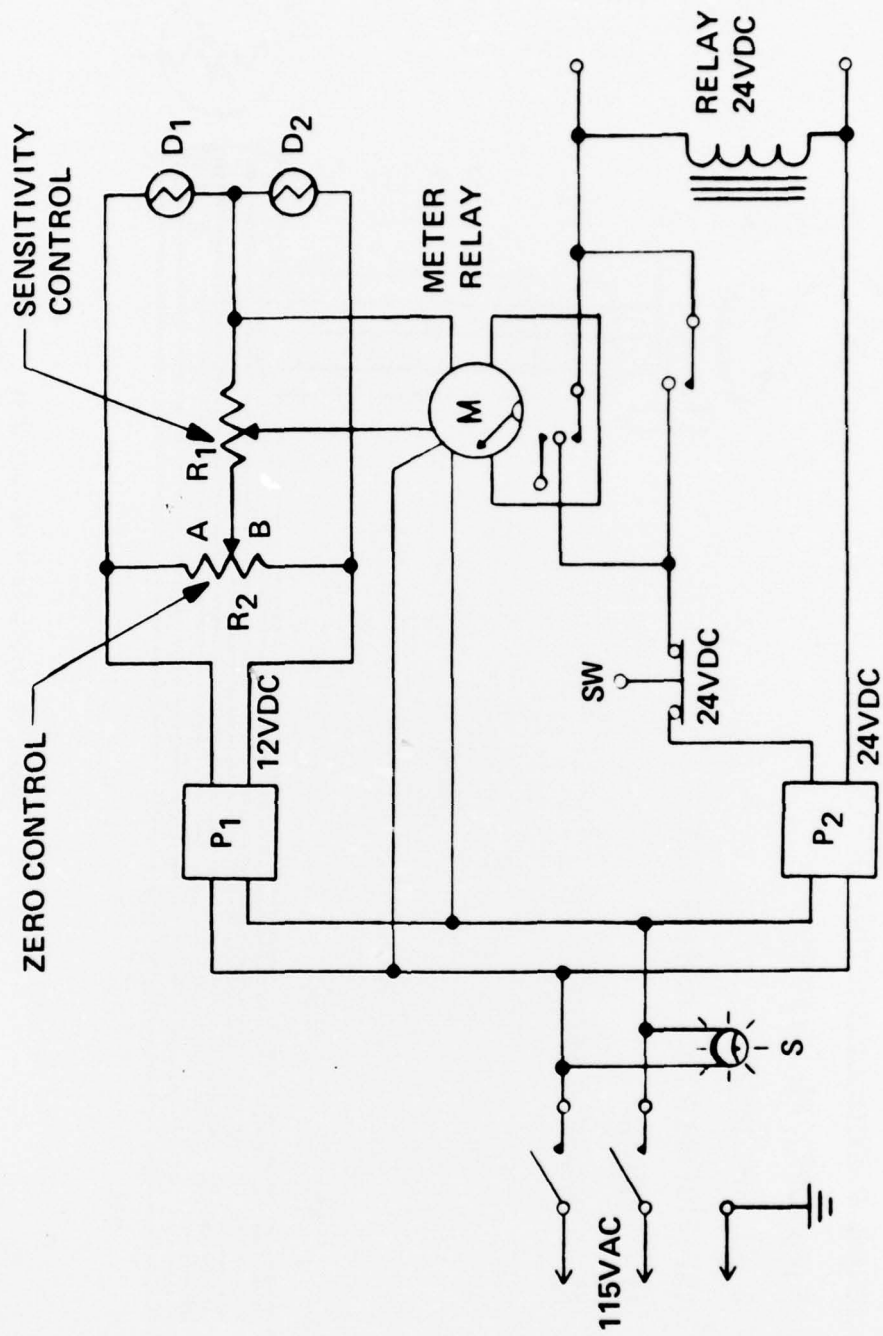


Fig 40 Schematic of electric circuit

- AUTOMATIC ON-LINE SULFURIC ACID ANALYZER
- MOLTEN EXPLOSIVES LEVEL MONITOR AND CONTROL SYSTEM
- MOLTEN EXPLOSIVES FLOW RATE MEASUREMENT SYSTEM
- COMPOSITION B ON-LINE ANALYZER SYSTEM

Fig 41 Instrumentation currently in development stage

- MODERNIZED AUTOMATED MFG OF ENERGETIC MATERIALS REQUIRES AUTOMATIC ON-LINE PROCESS INSTRUMENTATION
- AUTOMATIC ON-LINE INSTRUMENTATION HAS BEEN DEVELOPED TO MEASURE  $\text{HNO}_3$  /  $\text{HNO}_2$  /  $\text{H}_2\text{SO}_4$  AND SET POINTS IN THE CONTINUOUS TNT PROCESS



- AN AUTOMATIC MALFUNCTION DETECTION AND CONTROL SYSTEM HAS BEEN DESIGNED AND DEVELOPED FOR THE CONTINUOUS AUTOMATED MELT POUR LINE
- THE DEVELOPED INSTRUMENTATION HAS BEEN DESIGNED TO INTERFACE WITH EITHER ANALOG OR DIGITAL CONTROL SYSTEMS TO OPTIMIZE PRODUCTION
- INSTRUMENTAL DEVELOPMENT FOR APPLICATION TO PROCESS CONTROL OF COMP-B AND THE AUTOMATED MELT POUR LINE IS CURRENTLY IN PROGRESS

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